

# Quality Assurance Project Plan

EPA Region 5 Records Ctr.



280289

## **Remedial Investigation/ Feasibility Study Crab Orchard National Wildlife Refuge**

U.S. Fish and Wildlife Service  
U.S. Department of Interior  
Marion, Illinois  
and  
Sanganco-Weston, Inc.  
Atlanta, Georgia

May 1986



**O'BRIEN & GERE**



*Rich Boice*

**O'BRIEN & GERE**

May 28, 1986

Mr. Dick Ruelle  
U.S. FISH & WILDLIFE SERVICE  
1830 Second Avenue  
Rock Island, IL 61201

Re: Remedial Investigation (Feasibility  
Study) - Crab Orchard NWR  
Quality Assurance Project Plan

File: 3114.001

Dear Mr. Ruelle:

Enclosed is the Quality Assurance Project Plan (QAPP) for the Remedial Investigations being conducted at the Crab Orchard National Wildlife Refuge. The QAPP is inclusive of both the Phase I Sampling and Analysis which was completed in December 1985, and the Phase II Sampling and Analysis which will be undertaken after approval of this QAPP by the U.S. EPA, Region V.

If there are any questions or comments, please contact us at your convenience.

Very truly yours,

O'BRIEN & GERE ENGINEERS, INC.

*Steven R. Garver*  
for Steven R. Garver, P.E.  
Vice President

DRI:jld:D5:63

cc: Dr. C.B. Murphy, Jr.  
Mr. Richard Boice  
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Mr. John Hanson

QUALITY ASSURANCE PROJECT PLAN (QAPP)

REMEDIAL INVESTIGATION/  
FEASIBILITY STUDY  
CRAB ORCHARD NATIONAL WILDLIFE REFUGE

U.S. FISH AND WILDLIFE SERVICE  
U.S. DEPARTMENT OF INTERIOR  
MARION, ILLINOIS  
AND  
SANGAMO-WESTON, INC.  
ATLANTA, GEORGIA

MAY, 1986

O'BRIEN & GERE ENGINEERS, INC.  
1304 BUCKLEY ROAD  
SYRACUSE, NEW YORK 13221

REMEDIAL INVESTIGATION/FEASIBILITY STUDY  
(RI/FS)

REVISED QUALITY ASSURANCE PROJECT PLANT

Project Title: Crab Orchard National Wildlife Refuge  
EPA Project Officer: Richard Boice

Prepared by: O'Brien & Gere Engineers, Inc.

Date: 5/28/1986

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Date: 5/28/1986

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Date: 5/28/86

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Date: \_\_\_\_\_

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Date: \_\_\_\_\_

Approved: \_\_\_\_\_  
EPA QA Officer

Date: \_\_\_\_\_

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REFERENCES FOR ADDITIONAL DETAILS

1. Review of Previous Information (Jan. 1985)
2. RI/FS Work Plan (June 1985)
3. Work Plan Supplement - Phase I (Dec. 1985)
4. Work Plan Supplement - Phase II (April 1986)

## SECTION 1 - PROJECT DESCRIPTION

### 1.01 Introduction

O'Brien & Gere Engineers, Inc., is currently responsible for a Remedial Investigation/Feasibility Study (RI/FS) at the Crab Orchard National Wildlife Refuge in Marion Township, Williamson County, southern Illinois. This study encompasses thirty-three (33) specific sites, including two control sites, located on the Refuge. As required by the Environmental Protection Agency (EPA), a Quality Assurance Project Plan (QAPP) has been prepared for this RI/FS and is presented herein.

This QAPP presents, in specific terms, the policies, organization, objectives, activities and specific Quality Assurance (QA), and Quality Control (QC) activities designed to achieve the data quality goals of this project. Where possible, existing QA/QC guidelines, policies programs, etc., are incorporated into the QAPP by reference.

The purposes of this remedial investigation are: 1) to determine the nature and extent of any contaminant problem at several sites (Table 1) located around the eastern section of the Crab Orchard Lake, (Figure 1) on the Crab Orchard National Wildlife Refuge and tributaries that drain into Crab Orchard Lake and 2) to gather all data necessary to support the Feasibility Study. This will involve the following activities:

- Determine current groundwater gradients.
- Determine the extent of groundwater contamination that has occurred and the rate and direction of contaminant migration.



TABLE 1  
CRAB ORCHARD REFUGE  
SAMPLING SITES

<u>Site #</u>	<u>Type</u>	<u>Name</u>
<u>Group 1</u>		
3	Landfill	Area 11 South Landfill
4	Landfill*	Area 11 North Landfill
5	Pond	Area 11 Acid Pond
<u>Group 2</u>		
7	Surface Water	D Area SE Drainage
7A	Surface Soil	D Area North Lawn
8	Surface Water	D Area SW Drainage
9	Surface Water	P Area NW Drainage
10	Surface Water	Waterworks North Drainage
11	Surface Water	P Area SE Drainage
11A	Surface Soil	P Area North
20	Surface Water	D Area South
<u>Group 3</u>		
12	Landfill*	Area 14 Landfill
13	Surface Soil	Area 14 Change House Site
14	Surface Water	Area 14 Solvent Storage
<u>Group 4</u>		
15	Pond	Area 7 Plating Pond
16	Surface Soil	Area 7 Industrial Site
<u>Group 5</u>		
17	Landfill	Job Corps Landfill
<u>Group 6</u>		
18	Surface Soil	Area 13 Loading Platform
19	Surface Soil	Area 13 Bunker 1-3
30	Control*	Munition Control Site
<u>Group 7</u>		
21	Landfill	Southeast Corner Field
<u>Group 8</u>		
22	Surface Water	Old Refuge Shop
24	Surface Water	Pepsi-West
25	Surface Water	COC at Marion Landfill
26	Surface Water	COC below Marion STP
27	Surface Water	COC below I57 Dredge Area

TABLE 1  
(Continued)

CRAB ORCHARD REFUGE

SAMPLING SITES

<u>Site #</u>	<u>Type</u>	<u>Name</u>
<u>Group 9</u> 28	Landfill	Water Tower Landfill
<u>Group 10</u> 29	Landfill	Fire Station Landfill
<u>Group 11</u> 32 33	Landfill Surface Soil	Area 9 Landfill Area 9 Building Complex
<u>Group 12</u> 34	Lake	Crab Orchard Lake
<u>Group 13</u> 31	Control*	Refuge Control Site

- Assess levels of contaminated soil that may be present adjacent to disposal areas.
- Identify the areal extent of disposal areas.
- Identify specific contaminants which may pose acute or chronic hazards to public health, welfare or the environment.
- Identify pathways of contaminant migration from the sites.
- Define on-site physical features and facilities that could affect contaminant migration, containment, or cleanup.

O'Brien & Gere will furnish all personnel, materials and services necessary for or incidental to performing the remedial investigation on the Crab Orchard National Wildlife Refuge.

The remedial investigation consists of eight tasks:

Task 1 - Description of Current Situation

Task 2 - Investigation Support

Task 3 - Site Investigation

Task 4 - Preliminary Remedial Technologies

Task 5 - Site Investigations Analyses

Task 6 - Final Report

Task 7 - Community Relations

Task 9 - Additional Requirements

## 1.02 Site Location and History

Crab Orchard National Wildlife Refuge (CONWR or the Refuge) is located in southern Illinois primarily within Williamson County, but also extends into neighboring Jackson, Union and Johnson Counties. There are twelve lakes located within the Refuge including Crab Orchard

Lake. Crab Orchard lake was completed in 1940 and has a surface area of 6,965 acres, a maximum depth of 30 feet and 635 acre-feet of storage capacity. The watershed drainage area is 109,261 acres. In addition to supporting an active sport fishing population, the lake serves as water supply (approx. 280,000 gallons per day) for the Refuge and Federal Penitentiary located southeast of the Refuge. The City of Marion has a supplemental water intake in the Lake which has rarely been used.

The Refuge is administered by the U.S. Fish and Wildlife Service (FWS) of the Department of the Interior (DOI). During the early 1940s and continuing to the present, a number of industries have been active on the Refuge. Industrial activity was especially heavy during World War II when as many as 10,000 persons were employed by a number of defense-related industries. The section of the Refuge containing the industrial facilities lies within the eastern drainage area for the Crab Orchard Lake. The western portion of the lake has been used primarily for recreational purposes.

These industrial facilities were involved in a variety of manufacturing processes such as:

- Manufacture of land mines and bombs
- A munitions plant
- Manufacture of printing inks
- Production of radio speakers
- Metal plating, painting, metal work electrical work

To support these facilities, industrial dumps were developed within the Refuge. During the early 1940s, too, the Crab Orchard site was repeatedly sprayed with lead arsenate to control insects.

From the late 1970s through the present, sampling has been conducted to permit analysis of contamination. Until 1981, the main parameters of interest were lead, mercury, and other heavy metals, notably cadmium. After 1981, analyses were conducted also for PCBs, dioxins, and benzo furans.

Phase I of the Remedial Investigation encompassed thirty-three (33) sites, including two control sites. The histories of each site are as follows:

#### Site 3: Area 11 South Landfill

Areas 11 and 12 are currently abandoned sites of explosives and nitrogen fertilizer manufacturing as well as munitions loading. The Olin Corporation is reported to have operated a dynamite line there which was later reportedly sold to U.S. Powder. A number of fires and explosions are known to have occurred in these areas. Use of lead azide in the area is suspected. RDX may have been used in this area. Many of the buildings and grounds have been "torched" to remove residuals of flammable material. Most of the buildings are covered with a spark-retarding asbestos siding material. Also, within Area 11 are storage areas where explosive powders were stored in rubber-lined underground trenches. A burning pad is evident to the south of Area 11 where oil residues, 50-calibre powder magazines and small powder cylinders are noticeable on the surface. The evaluations of these areas are not included in this scope of work.

*See 11  
degrading*

The Area 11 South Landfill is located adjacent to what appears to be an old railroad bed. Much surface and buried litter is evident over

an area of perhaps 10 acres. In addition to railroad track, ties and ballast, the following were also observed: cinders and charred wood, powder canisters, piping, metal, mesh, bricks, pumice blocks, 30- and 55-gal drums, reinforcing bars, a laboratory flask and miscellaneous wire and plastic articles. One mound on the bank just above the stream bed has several of what appeared to be metal vents on the top and a 4-in stainless steel pipe drain extending from the bottom. The stream bed west of the road appeared to contain especially heavy concentrations of debris. Black tars and ash were evident in the stream bed.

#### Site 4: Area 11 North Landfill

The Area 11 North Landfill appears to have been the site of a large (2 to 3 acre) impoundment. The impoundment is flat in the middle and has small intermittent stream or marsh areas bordering the east and west boundaries. Water appears to flow from south to north following periods of precipitation. The reinforced concrete remains of a dam can be seen at the northwest end of the site. A large earth bunker is located immediately to the west. It may have been built with earth excavated from the semi-marshy lagoon area and may have been constructed to protect the explosives processing areas located further to the west. It was suggested that RDX or magnesium may have been stored underwater here or the area may have been used to detonate explosives or for experimental detonations. The level bottom of the impoundment shows a number of bare patches of fine white silt or clay. Other weathered areas showed horizontal layering of white and gray

sediments. A number of dynamite-type fuses were noticed here as well as a small powder carrier, 1.5-in dia by 3 in, with the fuse intact. Small lead chunks were also observed.

#### Site 5: Area 11 Acid Pond

The Area 11 Acid Pond is a diked impoundment approximately 300 ft x 150 ft which received drainage flowing north from the Area 11 process buildings. The dike extends 5 to 6 ft above the current water level. A 12 inch diameter pipe exits to the west through the levee to a valve box which controls the discharge from the pond to a small stream. This drainage then exits through the woods and swampy areas to the north. It is claimed that a spill of low-pH water (nitric acid) from the pond years ago killed all of the downstream vegetation for 1/4 mile. A large stand of dead trees is still visible along the creek north of the pond.

#### Sites 7, 8, 9, 10 and 11

##### D AREA SOUTHEAST DRAINAGE

##### D AREA SOUTHWEST DRAINAGE

##### P AREA NORTHWEST DRAINAGE

##### WATERWORKS NORTH DRAINAGE

##### P AREA SOUTHEAST DRAINAGE

The Olin D and P Areas are active Olin operations north of Crab Orchard Lake. Explosives are currently manufactured in the D Area while research and development is conducted in the P Area. It is likely that chemicals handled in the P Area are non-conventional or "exotic".

Universal Match also previously conducted operations here under contract to the DOD. Their operations ceased after a large explosion.

Sites 7, 8, 9, 10 and 11 are locations within various drainage channels leading from the Olin D and P Areas. These discharge to the Lake near the Refuge Waterworks.

#### Site 7A:D Area North Lawn

There is a large (about 3 acre) lawn located northwest of the active Olin D Area complex. It is claimed that barrels of chemicals were dumped on a knoll within this lawn. No evidence of a knoll was seen during the site visit, but a number (about 8) of depressed brown patches were evident on the lawn. A visually clean drainage channel is located south of the lawn and exits under the fence to the west. Other moist drainage areas extend to the wooded area to the west of the site.

#### Site 11A: P Area North

Located outside of the fence north of the Olin P Area is an abandoned L-shaped loading area with connecting covered walkways approximately 100 ft and 85 ft. The central structure contains a loading dock and a steamhouse containing a concrete pit with about 5 ft of clear standing water. An old roadbed runs west and north of the structure and draining swales surround all of the buildings. An abandoned (?) sewer line also runs across the north edge of the site. It has been reported that contaminants were dumped on the ground outside of the building.



#### Site 12: Area 14 Landfill

Area 14 was a site of munitions loading activity. Many of the buildings have been abandoned or demolished, but a few industries presently occupy some of the buildings. Historic aerial photos indicated what appeared to be landfill activity in the field east of the presently-occupied buildings. During the site visit the remains of a 100-ft dia circular impoundment were found at this site. The interior of the impoundment is presently overgrown with trees with trunk diameters of 8 to 10 in, indicating the date of the impoundment closure at about 1955 to 1965. The impoundment walls are about 6 ft high and the north wall has been breached to allow drainage to flow from the impoundment to an adjoining field. Several black oily pools are evident within and outside the basin. Other bare patches of black sediment and tars are located around the basin floor.

#### Site 13: Area 14 Change House Site

Southeast of the active Diagraph-Bradley buildings on Area 14 was an old building which was recently demolished. Formerly, it was the site of a "Change House" where workers changed their clothing after working in the adjacent bomb-loading buildings. At one time a company named CTI (Chemicals and Technology, Inc.??) manufactured explosives and other chemicals in this building. Other industries may also have occupied this building. The change building was supposedly located across from the bomb-loading building on a plot of land just southeast of the intersection of two roads on the north edge of a big dirt mound. The concrete floor of the change house is under this mound. Aerial

photos show another building (no longer present) further east of the corner; field inspection revealed several 1/2-in reinforcing rods imbedded in concrete near the corners of this building.

#### Site 14: Area 14 Solvent Storage

Diagraph-Bradley or Diagraph Marking Systems currently operates within a complex of buildings in Area 14. They produce inks, stencils, stencilboards and marking pens. Linseed oil and various solvents are handled in bulk and in drums here. Some of the bulk solvents noted were: T25 Xylene, T8 Diacetone Alcohol, T9 Diethylene Glycol, and T18 Methyl Cellosolve. Several compressed gas cylinders are also present. At least two drum storage areas containing 50 to 200 drums were also noted. Spill containment facilities are minimal. A drainage ditch runs north parallel to the road west of the buildings. Process water from the Diagraph-Bradley buildings enters this ditch from a standpipe.

*Perhaps it should say in CPC inspection*

#### Sites 15 and 16

##### AREA 7 PLATING POND

##### AREA 7 INDUSTRIAL SITE

Area 7 contains a complex of 33 identical buildings which have been used for a variety of industrial purposes during the past 40 years. Each of the six rows of buildings was previously served by a railroad siding.

Within a wooded rise to the south is located a small pond (approximately 50 ft x 30 ft) which is bermed about five ft above the current

water level. The current water depth is estimated to be about four ft. It is claimed that this pond was used to receive plating wastewaters from Olin operations which were located in this area at one time. PCBs, lead and other heavy metals may be of concern here.

Many of the buildings on the Area 7 site are used for dry warehousing purposes. However, two specific locations have been specified for sampling. Buildings 3-4, 3-5, and 4-4 are used by Pennzoil for waste oil recovery and recycling operations. Black residues are noticeable around some of these buildings. Buildings 5-2 and 5-3 are used by a refurbisher of mining machinery. Black residues are also evident around these buildings. A drainage channel runs from south to north through the center of the site.

#### Site 17: Job Corps Landfill

Northeast of the Refuge Waterworks is a small (approximately 10 acre) pond created by Job Corps workers in the mid-1960's. Attention has recently been brought to this pond because as many as thirty or more geese carcasses have been found floating on the water or littering the shores. Some of these carcasses have been relatively fresh while others were in various state of decay. The Fish and Wildlife Service has completed extensive analyses of these carcasses and has ruled out a variety of potential chemical causes. A definite conclusion has not yet been reached.

The "Job Corps" landfill was discovered while investigating the geese kills. It is located within a wooded area to the north and adjoining the pond and covers an area of perhaps an acre or more. It appears to be mainly surface litter dumped in spots and perhaps spread

around, although deeper spots cannot be ruled out. Many of the surface articles appear to be connected with food preparation, e.g. institutional-size food cans, and a variety of bottles. The bottle styles and labels suggest a date of the mid-1950's, which was consistent with a 1956 Illinois automobile license plate also found. Many of the debris piles are overgrown by thick brush. Two bare patches (less than 6-ft diameter each) were located among the debris. Mica flakes and small electrical contacts were found in one of these. It is claimed that small electrical capacitors were also found here, but none were noted during this site visit. Probing with a trowel revealed no further debris beneath the top inch of soil.

#### Site 18: Area 13 Loading Platform

On the northwest end of the Area 13 munitions storage bunkers is a concrete loading platform adjacent to the abandoned and dismantled rail line. It is reported that munitions-type chemicals were dumped off the platform. The site inspection indicated that the elevated concrete loading dock is about 235 ft long by 10 ft wide and about 5 ft high. The dock is supported on concrete posts spaced 9 ft apart. The northwest side contains stone bedding (probably from the oil railroad bed) with a number of small areas of ponded water. No unusual vegetation changes were detected. The only unusual item was a pile of dirt and stone rubble off the west end of the dock with a rusted drum shell nearby.

#### Site 19: Area 13 Bunker 1-3

Area 13 contains approximately 85 bunkers which were originally built for storage of 500-lb bombs. Most of them still contain explosives, leased mainly to Olin and U.S. Powder. Agricultural fields are cultivated between the bunkers. Formerly, they were fruit orchards.

It has been reported that chemicals were poured out near Bunker 1-3, probably in the field next to it. A site inspection did not reveal any significant signs of impact. Evidence of fill activity (scattered red bricks) is widespread. An L-shaped area of brown vegetation difference was noted to the west side of the bunker.

#### Site 20: D Area South

An abandoned building is located within the fenced southeastern end of the Olin D Complex. It was reported that chemicals were dumped here. A drainage swale originating at the building runs east outside of the fence. A four-in pipe (dripping) extends from the Olin Area under the fence and discharges to this ditch. A slight sheen was noticeable on the surface water in pooled areas of the ditch.

#### Site 21: Southeast Corner Field

At the southeast corner of the refuge is a field which is thought to be the site of a very old landfill. A pile of concrete pieces, possibly from an old bridge, is located immediately inside the fence. The topography gradually slopes to the south and east with a swampy drainage ditch at the bottom of the slope. No other evidence of debris

could be found. Trees as large as 24-in in diameter suggest that the area has not seen any soil-disturbing activity within the past 60 to 70 years.

#### Site 22: Old Refuge Shop

North of the refuge along Wolf Creek Road is the old refuge headquarters, now leased by Diagraph Bradley. Behind this building is located the old shop area of the refuge. Pine poles were treated here with pentachlorophenol and shipped to various spots around the country. Outside the fence to the north is a small pool which receives drainage from the old shop area. The pool contains a green-yellow scum and drains through the woods to the northwest.

#### Site 24: Pepsi-West

The Pepsi Cola Bottling Company in Marion could potentially discharge to Crab Orchard Creek. It is not known whether the City or State monitor environmental activities here. A site inspection indicated that it was unlikely that discharges issued directly south to the Creek, since the entire south end of the property rises 4 to 8 ft in elevation above the parking lot. Drainage ditches, however, were located to the north adjacent to the street. These probably receive surface runoff only.

#### Site 25: Crab Orchard Creek at Marion Landfill

The old Marion landfill is off Old Creal Springs Road and directly abuts Crab Orchard Creek. It has apparently been inactive for a

number of years. A visible face of trash can be seen by travelling upstream several hundred yards from the road. Near to this is a small pond (approximately 3/4 acre).

Sites 26 and 27:

CRAB ORCHARD CREEK BELOW MARION STP

CRAB ORCHARD CREEK BELOW 157 DREDGE AREA

The Marion sewage treatment plant discharges to Crab Orchard Creek somewhere upstream of Court Street. A number of samples downstream from the Marion STP are scheduled to assess the quality of various stretches of Crab Orchard Creek.

Site 28: Water Tower Landfill

Aerial photos indicate landfilling activities adjacent to the water tower near Areas 7 and 14. These activities are not visually apparent today. The sloping face northeast of the water tower is heavily overgrown with briars and rutted with several major gullies. Only a small amount of refuse is evident on this slope. A previous soil sample taken in this area showed 800 ppm lead concentration. More activity is evident in the woods at the bottom of the slope. A number of rusted drums, metal parts and tar residues can be found here. Standing water in the main drainage gully shows a slight sheen on the surface. Several small mounds are within the woods and a larger mound is located at the top of the hill.

Site 29: Fire Station Landfill

Located southwest of the refuge fire station is a large field which was used for storage of mining machinery until several years ago. The northern and western edges of this field show evidence of a large dump site. Debris is evident on the face which drops 4-5 ft. to a swampy area to the west. Previous sampling near an evergreen tree on the north side showed lead concentrations of 553 ppm. A slight sheen is noted in spots within the swamp. Most of the debris consists of concrete, metal, wire and other machinery-related items. It was reported that Olin dumped heavily here and there once was a very hot fire. Ignitable magnesium is suspected to be in the fill. An empty 30-gal drum labelled "Magnesium Powder" was found along the south portion of the eastern face.

*See Ref. material*

Site 30: Munition Control Site

A munition control site is established on an area where the operations involved only ammunitions manufacture.

Site 31: Refuge Control Site

A control sampling station is established on an uncontaminated area of the refuge behind the new Refuge headquarters. Selection of the control site was coordinated with the Refuge Manager, following a site visit.



Site 32: Area 9 Landfill

*La. 9005 Pump*

The Area 9 Landfill was used during the 1950's and early sixties and was probably closed in 1964. The Landfill is located below approximately 100 yds south of Crab Orchard Lake and approximately 100 yards east of the building complex. Runoff can drain from the landfill into an intermittent creek and then to the Lake. The limits of the landfill are discernible by changes in the topography and vegetation. It is approximately 2.5 acres with a fill thickness of 8 to 10 feet in the middle and 6 feet at the edges. Waste materials are exposed at locations where cover material has eroded. Some areas are void of vegetation.

The volume of the landfill is estimated to be from 16,000 to 35,000 cubic yards. Materials visible on the surface appear to be electrical components consisting of small capacitors, capacitor parts, large chunks of a golden resin, and a large number of 3-inch steel cuplike pieces.

Wastes were burned, compacted in a swale and covered when the landfill was active. Specific compounds of concern include lead, acetate, PCBs (Aroclor 1254 and 1242), and PCB burning products. Other possible materials from capacitor manufacturing include mica, silver, cyanide, aluminum hydroxide, aluminum oxide, gold, copper, zinc, hydrochloric acid, styrene, nitric acid, phosphoric acid, and borates. Other industrial wastes may include cyanides, printing inks and lead-based explosives. A magnetometer survey indicated a high concentration of metals on the east side of the landfill.

### Site 33: Area 9 Building Complex

The Area 9 Building Complex was leased during the period from 1946 to 1962 as the Ordill Facility containing the Sangamo Capacitor Division. Manufacturing operations began in the early 1950's. This division manufactured power factor capacitors, AC motor run capacitors, and a variety of DC capacitors. The components were of various types and included aluminum, electrolytes, mica, and silver and lead foil. The Division also manufactured small transformers that used mineral oil as a dielectric.

Subsequently, Olin Corporation started using the industrial facilities at the site. Olin manufactured explosives that were used to start jet engines. The company used nitro-glycerine in its operation.

### Site 34: Crab Orchard Lake

Crab Orchard Lake (completed in 1940) has a surface area of 6,965 acres, a maximum depth of 30 feet, and 635 acre-feet of storage capacity. The watershed drainage area is 109,261 acres. The lake has a retention time of approximately 0.8 years. Water enters the lake through several creeks, including Crab Orchard Creek on the eastern end of the lake and an intermittent creek adjacent to the Area-9 Land-fill. Water leaves the lake through Crab Orchard Creek on the western end of the lake. In addition, 280,000 gallons/day of water is used by the Refuge.

The eastern section of the lake is near several manufacturing operations established since the 1940s.

### 1.03 Project Objectives

The primary objective of the RI/FS is <sup>determine hazards to human health and to the environment and to</sup> recommend the most cost-effective source control and off-site remedial actions. Source control remedial actions include measures to prevent, reduce, or eliminate contamination either by containing the hazardous wastes in place or removing them from the site. Off-site remedial actions include measures to mitigate the effects of hazardous waste contamination that has migrated beyond the site. Appropriate source control and off-site remedial actions will be formulated and analyzed in detail after sufficient data have been generated through the remedial investigation.

Based upon existing data, remedial actions that may be appropriate for the CONWR site include, but are not limited to, one or a combination of the following:

- No action.
- Removal and disposal of waste material.
- Solidification or stabilization of waste material.
- In place reconstruction or encapsulation of waste material.
- Continued off-site monitoring.
- Limit access to contaminated areas.
- Groundwater collection and treatment systems.
- Surface water drainage measures to prevent ponding on or near sites of contamination.
- Construction of groundwater barriers.
- Construction of a clay or synthetic cap over contaminated.
- *Contaminated soil incineration.*

Presently, the available data and information on the site are insufficient to allow a definitive selection, screening, and feasibility study of remedial action alternative.

#### 1.04 Project Description

The remedial investigation/feasibility study (RI/FS) for the Crab Orchard National Wildlife Refuge Site is intended to determine the nature and extent of contamination, to develop and evaluate remedial alternatives and to identify cost-effective remedial actions to be taken at contaminated sites on the refuge which reduce risks to acceptable levels. To accomplish this, the following tasks will be completed:

- characterize the on-site soil, sediment, water and biological samples for the presence of hazardous contaminants (includes landfill, surface soil, pond and lake water).
- identify pathways of chemical migration from the site.
- characterize the off-site soil, sediment, water and biological samples for key hazardous components.
- determine and describe on-site physical features that could affect migration of key hazardous components, methods of containment, or methods of remedial action clean-up.
- develop viable remedial action alternatives.
- permit the evaluation of the remedial action alternatives.
- recommend the most cost-effective technically feasible remedial option which has the ability to reduce impacts on human health, welfare and the environment to an acceptable level.
- prepare a conceptual design of the recommended remedial action alternative.

## TASK 1 - DESCRIPTION OF CURRENT SITUATION

O'Brien & Gere will describe the background information pertinent to the sites and outline the purpose and need for remedial investigations at those locations. The data gathered during any previous investigations or inspections and other relevant data will be used. A partial list of sources on published and unpublished data available on Crab Orchard Creek watershed and Crab Orchard Lake is included in the Work Plan Supplement (December 1985).

The sub-tasks will include site background, nature and extent of the problem at the sites under investigation and a history of response actions.

## TASK 2 - REMEDIAL INVESTIGATION SUPPORT

Prior to initiating any field investigations, the following preliminary work will be completed.

### A. Site Visit

Initial site visits will be conducted to become familiar with site topography, access routes, and proximity of receptors to possible contamination, and collect data to support the Site Health and Safety Plan. Site surveys will be conducted to identify and stake boundaries of known contaminated areas, monitoring wells, and soil borings, and to identify sediment sample locations. A geophysicist will evaluate the applicability of using geophysical methods to determine the existence of contaminant groundwater plumes if necessary. The visit will

be used to verify the site information developed in Task 1. The Site Health and Safety Plan will be amended, if necessary, as a result of this visit.

B. Site Maps

O'Brien & Gere will prepare site maps showing all wetlands, water features, drainage patterns, tanks, buildings, utilities, paved areas, easements, right-of-ways, and other features. The site maps and all topographic surveys will be of sufficient detail and accuracy to locate and report all existing and future work performed at the sites. Areas to be investigated will be mapped using existing topographic maps or aerial photos. After the initial analytical data have been reviewed and where necessary for remedial efforts, the topographic maps will be prepared with 1-foot contours referenced to the National Geodetic Vertical Datum with a scale of 1 inch to 50 feet. The maps will extend 200 feet beyond site boundaries and include all drainages to Crab Orchard Lake.

Boundary lines encompassing contaminated areas will be identified. The boundary lines for the landfill study sites will be identified using magnetometer and electromagnetic methods. The boundary conditions will be set so that subsequent investigations will cover the contaminated media in sufficient detail to support the feasibility study. The boundary conditions may also be used to identify boundaries for

site access control and site security. If necessary, a fence or other security measures may be installed as an initial remedial measure.

C. Dispose of On-Site Generated Waste

All wastes generated by on-site activities will be labelled, drummed and stored within controlled-access areas. Wastes which will be drummed include: all drill cuttings, all purged groundwater from well development, decontamination wash water and disposable protective clothing. These materials, if contaminated, will be properly disposed of during cleanup actions as identified by the feasibility study.

TASK 3 - SITE INVESTIGATIONS

O'Brien & Gere will conduct remedial investigations necessary to characterize the site and its actual or potential hazard to public health and the environment. The site investigations will generate data of adequate technical content to support detailed evaluations of alternatives during the feasibility studies.

The site investigations will be conducted in two phases. Phase I *has been completed* will include geophysical surveys, hydrogeologic investigations, installation of groundwater monitoring wells, and a screening of each site to analyze composited samples for a broad array of potential contaminants as listed in Table 2. Selected samples will be confirmed by a full analysis for the priority pollutants.

TABLE 2  
RI/FS ANALYTICAL PARAMETERS

1. Purgeable Priority Pollutants  
(Screening and Full Analysis)
2. Acid Extractable Priority Pollutants  
(Screening and Full Analysis)
3. Base/Neutral Extractable Priority Pollutants  
(Screening and Full Analysis)
4. Pesticide/PCB Priority Pollutants  
(Screening and Full Analysis)
5. PCB's
6. Metals
  - ICP scan
  - Priority Pollutant Metals by AA Spec
  - Mercury
7. EP Toxicity
8. Cyanide 40
9. Indicators
  - pH (field)
  - Specific Conductance (field)
  - Total Organic Carbon
  - Total Organic Halogens
10. Explosives Residues by HPLC
11. Nitrogen Series: TKN, NH<sub>3</sub>N, NO<sub>3</sub>N
12. PCDD/PCDF  
(Screening and Full Analysis)
13. Cation Exchange Capacity
14. Total Phosphorus
15. Primary and Secondary Drinking Water Standards
16. Percent Solids (for soil/sediments)



*sb 1*

The sites listed in Table ~~2~~ fall under five categories.

1. Landfills
2. Surficial Contaminant Sites
3. Streams
4. Ponds
5. Lake

Phase II will consist of additional sampling and analysis to fill in data gaps identified in Phase I and further assess the extent of contamination at each site where materials of concern are found. The general rationale in developing sampling and analysis schedules for each category of sites is shown in Table 3.

The sub-tasks under site investigations include:

- A. Geophysical Surveys
- B. Hydrogeologic Investigations
- C. Groundwater Sampling and Analysis
- D. Soil Investigation
- E. Surface Water and Sediment Sampling and Analysis
- F. Fish Sampling and Analysis

A. Geophysical Surveys

Geophysical investigations will be conducted to determine the extent of soil and groundwater contamination, if any, in the vicinity of several specified study sites as outlined in Appendix B. In particular, the geophysical investigations will be conducted at areas of suspected landfill activities, and will consist of magnetometer and electromagnetic induction (EM) surveys.

TABLE 3

REMEDIAL INVESTIGATION SAMPLING AND ANALYSIS SEQUENCE

<u>Site Category</u>	<u>Recon.</u>	<u>Phase I</u>	<u>Phase II</u>	<u>Contingency</u>
Landfills	Geophysics	Cores - depth composites - screening & full priority pollutants & explosives residuals + ICP metals - Install wells-analyze indicators + metals.	Radial & depth cores and wells for priority pollutants & explosives residuals found in cores & AA metals.	
Surface	Geophysics - locate utilities	Surf. Soils - screening & full priority pollutants and explosive residuals + ICP metals.	Depth soils Radial soils - surf. & depth Runoff - water & sediments & depth profile	
Streams - Waters - Sediments		Upstream/downstream - screening & full priority pollutants & explosive residuals Surf. sed: 2 near shore, 1 near lake - screening & full priority pollutants + expl. + ICP metals	Surf seds - int + depth sed. - priority pollutants found + AA metals	
Ponds - Waters - Sediments - Groundwater		(Same rationale as streams) (Same rationale as streams) Upgradient/downgradient wells (2) - indicators	Depth profile on sediments priority pollutants + expl. found in waters or sed.	Additional wells
Lake - Waters - Sediments - Biota		5 sites; primary & secondary - Drinking Water stds. (None) Sample & freeze	5 biota sites + 5 use sites: anything found in Phase I 5 sites: parameters found in Phase I parameters found in Phase I	
Control Sites - Lake control - Soil & groundwater control - Clean area - Munitions area		(All analyses included at other sites)	Full scans	

\*ICP: Metals analysis by Induced Coupled Plasma Spectrophotometry

AA: Metals analysis by Atomic Adsorption Spectrophotometry

B. Hydrogeologic Investigations

The hydrogeologic investigation will be used to determine the present and potential extent of groundwater contamination, if any, and evaluate the suitability of the site for on-site waste containment. Efforts will begin with a survey of previous hydrogeologic studies and other existing data (completed as part of Task 1 a and c). The survey will address the degree of hazard, the mobility of chemicals considered, the soil attenuation capacity and mechanisms, discharge/recharge areas, regional flow direction and quality, and effects of any pumping alternative. Subsequent to the survey of existing data, sampling programs will be developed to determine the horizontal and vertical distribution of chemicals considered and predict the long-term disposition of such chemicals.

C. Sampling and Analyses of Groundwater

Groundwater monitoring wells <sup>were</sup> ~~will be~~ installed during the Phase I sampling effort and <sup>will be</sup> sampled during Phase II. Additional monitoring wells will be installed, if necessary after existing on-site wells are sampled and the water analyzed for contaminants of concern. Then, based on the geophysical results (Task 3a) and results of contaminant analyses, the extent and scope of any additional hydrogeologic investigation will be determined.

D. Soil Investigation

O'Brien & Gere will develop and conduct a program to identify the location and extent of surface and subsurface soil, and sediment contamination. This process may overlap with certain aspects of the hydrogeologic study, e.g., characteristics of soil strata are relevant to both the transport of contaminants by groundwater and to the location of contaminants in the soil. These soil samples and an additional number of soil borings will be collected for analysis from various sampling sites around the refuge.

E. Surface Water and Sediment Investigation

O'Brien & Gere will develop and conduct a program to determine the extent of water and sediment contamination on selected refuge lakes, marshes, ponds and streams. This process may overlap with the soil investigation; data from lake sediments sampled may be relevant to surface water quality. A survey of existing data on surface water quality and quantity may be a useful first step.

F. Fish and Wildlife Investigations

Selected species of fish and other aquatic organisms on the refuge will be collected by FWS and analyzed by O'Brien & Gere for residual levels of contaminants previously identified in landfills and other contaminated areas on the refuge.

#### TASK 4 - PRELIMINARY REMEDIAL TECHNOLOGIES

##### A. Post-Investigation Evaluation

Either during or following the site investigations, O'Brien & Gere will assess the investigation results and recommend preliminary remedial technologies best suited to specific contaminant problems for each site. They will provide the basis for developing detailed alternatives needed for the completion of the feasibility studies. The data generated during the remedial investigations will generally be limited to accomplish the following:

1. Recommend types of remedial technologies appropriate to physical and site contaminant conditions.
2. Recommending whether or not to remove some or all of the waste for off-site treatment, storage, or disposal.
3. Determine the compatibility of groups of wastes with other wastes and with materials considered as part of potential remedial action. Recommend alternatives for treatment, storage, or disposal for each category of compatible waste.

#### TASK 5 - SITE INVESTIGATIONS ANALYSIS

The results of Tasks 1 through 4 will be used to prepare a thorough analysis and summary of all site investigations. The objective of this task is to ensure that the investigation data are sufficient in quality and quantity to support the feasibility studies.

The results and data from all site investigations will be organized and presented logically. The geographic groupings listed on Table 1 will form the basic structure for all of the assessments. This will permit the assessment of transport modes and impact to receptors.

A. Data Analysis and Endangerment Assessment

The site investigation data will be analyzed to develop a summary of the type and extent of contamination at the sites. The summary will describe the quantities and concentrations of specific chemicals at each site and ambient levels surrounding the sites. Ambient samples will be collected from control sites.

Data collected during the RI phase will also be evaluated to determine if environmental conditions or materials at the site present potential hazards to human health or welfare, or to the environment. Existing standards will be reviewed to help formulate conclusions and recommendations regarding the hazard potential of the site. If additional hazards are identified, the risks associated with each hazard will be summarized.

This analysis will discuss the degree to which either source control or off-site measures are required to significantly eliminate the threat, if any, to public health or the environment. If the results of the investigation indicate that no threat or potential threat exists, a recommendation of no remedial response will be made.

*to be included in the assessment.*

A technical memorandum will be prepared by the Respondents summarizing the hazard evaluation process and presenting the results of the hazard assessment.

#### TASK 6 - FINAL REPORT

A final RI report will be prepared to consolidate and summarize the data collected during the RI. The report will include a discussion of the data acquired during the RI and the hazard identification and risk potential of the contaminants detected. Ten copies of the remedial investigation report will be submitted to the FWS. The report will be structured to enable the reader to cross-reference with ease.

#### TASK 7 - COMMUNITY RELATIONS

The Community Relations program is included as Task 7; however, the dissemination of information to the public will be coordinated by the FWS throughout the duration of the study. O'Brien & Gere will provide personnel, at the Service's discretion, to support the programs as community relations must be integrated closely for all remedial response activities.

The objectives of this effort are (1) to keep the community informed as to the study progress, (2) to achieve community understanding of the actions taken, and (3) to obtain community input, and support prior to selection of the remedial alternative(s).

## TASK 8 - ADDITIONAL REQUIREMENTS

### A. Reporting Requirements

O'Brien & Gere will prepare monthly reports to describe the technical and financial progress of the project. These reports will discuss the following items:

1. Identification of sites on which activity took place and the nature of those activities.
2. Status of work at the site and programs to date.
3. Percentage of completion.
4. Difficulties encountered during the reporting periods.
5. Actions being taken to rectify problems.
6. Activities planned for the next month.
7. Changes in personnel
8. A comparison of target and actual completion dates for each element of activity including project completion and an explanation of any schedule deviations in the work plan.
9. Progress Reports on Items 1 through 8 will be submitted to FWS, who shall in turn relay them to USEPA and IEPA.
10. A Work Plan that includes a detailed technical approach and schedules will be submitted for the proposed feasibility study.



B. Site Health and Safety Plan

Prior to conducting any field activities O'Brien & Gere will provide any necessary modifications to the Site Health and Safety Plan as presented in Appendix C. The plan is consistent with:

Section 111(c)(6) of CERCLA.

EPA Order 1440.3 - Respirator Protection

EPA Order 1440.2 - Health and safety requirements for employees engaged in field activities.

EPA Occupational Health and Safety Manual.

Other EPA guidance as provided.

State Safety and health statutes.

Site conditions.

EPA Interim Standard Operating Safety Guide (September 1982) and applicable OSHA standards.

C. Quality Assurance/Quality Control (QA/QC)

O'Brien & Gere has prepared a Quality Assurance Project Plan (QAPP) for the sampling, analysis, and data handling aspects of the remedial investigation which is presented in Appendix A. The QAPP plan is consistent with U.S. Fish and Wildlife Service, State and Federal EPA requirements. The plan addresses the following points:

1. QA Objectives for Measurement Data, in terms of precision, accuracy, completeness, representativeness and comparability.
2. *Sampling Procedures.*
3. Sample Custody.
4. Field Equipment, Calibration Procedures, References and Frequency.
5. Internal QC Checks and Frequency.
6. QA Performance Audits, System Audits, and Frequency.
7. QA Reports to Management.
8. Preventative Maintenance Procedures and Schedule.
9. Specific Procedures to be used to routinely assess data precision, representativeness, comparability, accuracy, and completeness of specific measurement parameters involved. This section will be required for all QA project plans.
10. Corrective Action.

D. Site Sampling Plan

Site specific sampling plans for Phases I and II of site investigations have been developed for this Remedial Investigation, and are summarized in Section 1.05 of this QAPP. The sampling plan covers the sampling efforts described in the Remedial Investigation work plan and addresses the following topics:

- Sample types and tentative locations
- Sample equipment and procedures
- Sample handling, custody procedures, and preservation
- Sample documentation
- Sample shipping
- Analytical arrangements (scheduling)
- Analytical procedures
- QA/QC review procedures of data
- Analytical review of data
- Disposal of unused samples

#### 1.05 Sampling and Analysis

Phase I sampling and analysis details are set forth in Appendix B of the Site Sampling Plan dated 6/85. Additional details are included in the Work Plan Supplement dated 12/85. The Phase II sampling program is presented in detail in the Work Plan Supplement, Phase II Site Operations Plan dated 4/86.

Sampling activities under various Remedial Investigation Tasks are shown in Table 4. A listing of individual samples scheduled for Phases I and II sampling and analysis are included as Attachment 1. The parameters included in the various Analysis Sets are given in Table 5. The number of samples scheduled are summarized by sites and analysis sets in Table 6 for Phase II and Table 7 for Phase I. The rationale for Phase I sampling at these sites are indicated for each sample in Attachment 1 and explained in Attachment 2.

TABLE 4

SUMMARY OF ANALYSES TO BE PERFORMED

Task No. (WORK PLAN)	No. Samples Collected	No. For Screening	Full Analyses	Selected Parameters	No Spikes	No Dup(s)	Field Analyses	Comments
2-B Site Maps	--	--	--	--	--	--	--	1"=50' Scale with 1' contours
3-A Geophysical Survey	6 sites 6 sites	--	--	--	--	--	Terrain Conductivity Magnetometer	EM-31 Meter Used Proton Magneto- meter <i>EPA</i>
3-B Hyrdogeologic Investigations	9 wells to be installed	--	--	--	--	--	Fike Sta. - 4 wells Acid Pond - 1 well Refuge control-1 well Munciation Control - 1 well Water Tower - 2 wells	2" ID <i>SS</i> PVC Casing and well screening
3-C Groundwater Sampling and Analyses	16	--	5(I)	1(M) 4(Q) 6(S)			Temp, pH and Spec. Conditions	Samples will be collected and Analyzed in Phase II
3-D Soil Investigation	328	306	6-(F) 7(G) 9(H)	--	20	39		
3-E Surface Water and Sediment Investigation	36 71	26 48	10(I) 10(F) 2(H) 3(G) 8(I)	--	1 4	2 11		
3-F Biota	30	--	2(H)	28(T)	--	--	Length and Weight	Samples Frozen before shipping

Note:

The letters in parenthesis under full analysis and selected parameters indicate analysis sets (see Table 5).

Table 5 pg.1

PARAMETERS		ANALYSIS SET								
		A	B	C	D	E	F	G	H	I
1. Purgeable Priority Pollutants	-Screen	x	-	-	x	-	-	-	-	-
	-Full Anal.	-	-	-	-	-	x	x	x	-
2. Acid Extract. Priority Pollutants	-Screen	x	-	-	x	-	-	-	-	-
	-Full Anal.	-	-	-	-	-	x	x	x	-
3. Base/Neutral Extact. Prior. Poll.	-Screen	x	-	-	x	-	-	-	-	-
	-Full Anal.	-	-	-	-	-	x	x	x	x
4. Pesticide/PCB Priority Pollutants	-Screen	x	-	-	x	-	-	-	-	-
	-Full Anal.	-	-	-	-	-	x	x	x	x
5. PCB's		-	x	x	-	-	-	-	-	-
6. Metals - ICP Scan	-Screen	x	-	-	x	-	-	-	x	x
- Prior. Poll. scan by AA	-Full Anal.	-	-	-	-	-	-	-	-	-
- Mercury		x	-	-	x	-	-	-	x	x
- Cadmium		-	-	-	-	-	-	-	-	-
- Chromium		-	-	-	-	-	-	-	-	-
- Magnesium		-	-	-	-	-	-	-	-	-
- Lead		-	-	-	-	-	-	-	-	-
7. EP Toxicity - Chromium		-	-	-	-	-	-	-	-	-
- Cadmium, Chromium, Lead		-	-	-	-	-	-	-	-	-
8. Cyanide 40		x	-	-	x	-	-	-	x	x
9. Indicators - pH (field)		x	-	x	x	-	-	-	x	-
- Specific Conductance (field)		x	-	x	x	-	-	-	x	-
- Total Organic Carbon		x	-	x	x	-	-	-	x	-
- Total Organic Halogen		x	-	x	x	-	-	-	x	-
10. Explosives Residues by HPLC		x	-	-	x	-	-	-	x	-
11. Nitrogen Series: TKN, NH3, NO3		x	-	x	x	-	-	-	x	-
12. PCDD/PCDF	-Screen	-	-	x	x	-	-	-	-	-
	-Full Anal.	-	-	-	-	-	-	x	x	-
13. Cation Exchange Capacity		-	-	x	-	-	x	x	-	-
14. Total Phosphorus		x	-	-	x	-	-	-	x	-
15. Primary & Secondary Drinking Water Stds.		-	-	-	-	x	-	-	-	-
16. Percent Solids (on soil/sed only)		x	x	x	x	-	x	x	x	x

NOTE: SETS F & G are full analysis of parameters screened in SETS A & D resply.

SET H is full analysis of selected samples instead of SET D

SETS I to T are for samples analyzed in Phase II

Print range: E4.M52 & N4.X52; Y4.A654 & A4.A654; A54.A654 & B4.BL54; B44.BU54 & BV4.CF56

Table 5 pg.2

PARAMETERS		ANALYSIS SET (contd.)										
		J	K	L	M	N	O	P	Q	R	S	T
1. Purgeable Priority Pollutants	-Screen	-	-	X	-	-	-	-	-	-	-	-
	-Full Anal.	X	-	-	-	-	X	-	-	-	-	-
2. Acid Extract. Priority Pollutants	-Screen	-	-	X	-	-	-	-	-	-	-	-
	-Full Anal.	X	-	-	-	-	X	-	-	-	-	-
3. Base/Neutral Extract. Prior. Poll.	-Screen	-	-	-	-	-	-	-	-	-	-	-
	-Full Anal.	-	-	-	-	-	-	-	-	-	-	-
4. Pesticide/PCB Priority Pollutants	-Screen	-	-	-	-	-	-	-	-	-	-	-
	-Full Anal.	-	-	-	X	-	-	-	-	-	-	-
5. PCB's		-	-	-	-	-	-	X	X	-	-	X
6. Metals - ICP Scan	-Screen	-	-	-	-	-	-	-	-	-	-	-
- Prior. Poll. scan by AA	-Full Anal.	-	-	-	-	-	-	-	-	-	-	-
- Mercury		-	-	-	-	-	-	-	-	-	-	X
- Cadmium		-	-	-	-	-	-	X	X	-	-	X
- Chromium		-	-	-	X	-	-	-	-	-	-	-
- Magnesium		-	-	-	-	-	X	-	-	-	X	-
- Lead		-	-	-	-	-	-	X	X	-	X	X
7. EP Toxicity - Chromium		-	-	-	-	X	-	-	-	-	-	-
- Cadmium, Chromium, Lead		-	-	-	-	-	-	-	-	X	-	-
8. Cyanide 40		X	-	-	-	-	-	-	-	-	-	-
9. Indicators - pH (field)		-	-	-	-	-	-	-	-	-	-	-
- Specific Conductance (field)		-	-	-	-	-	-	-	-	-	-	-
- Total Organic Carbon		-	-	-	-	-	-	-	-	-	-	-
- Total Organic Halogen		-	X	-	-	-	-	-	-	-	-	-
10. Explosives Residues by HPLC		-	-	-	-	-	-	-	X	-	-	-
11. Nitrogen Series: TKN, NH3, NO3		-	-	-	-	-	-	-	-	-	-	-
12. PCDD/PCDF	-Screen	-	-	-	-	-	-	-	-	-	-	-
	-Full Anal.	-	-	-	-	-	-	-	-	-	-	-
13. Cation Exchange Capacity		-	-	-	-	-	-	-	-	-	-	-
14. Total Phosphorus		-	-	-	X	-	-	-	-	-	-	-
15. Primary & Secondary Drinking Water Stds.		-	-	-	-	-	-	-	-	-	-	-
16. Percent Solids (on soil/sed only)		X	X	X	X	X	X	X	X	X	X	X

SET H is full analysis of selected samples instead of SET D

SETS I to T are for samples analyzed in Phase II

## PHASE I SAMPLING &amp; ANALYSIS SUMMARY

(Revised March 10, 1986)

Table 6 pg.1

SITE NO.	SAMPLE TYPE	WATER NO.OF ANAL. SAMPL TYPE	WELL NO.OF ANAL. SAMPL TYPE	SOILS NO.OF ANAL. SAMPL TYPE	SEDIMENTS NO.OF ANAL. SAMPL TYPE	BIOTA NO.OF ANAL. SAMPL TYPE
3 AREA 11 SOUTH LANDFILL		0 -	0 -	3 A 1 F	1 A 1 D	0 -
4 AREA 11 NORTH LANDFILL		0 -	0 -	1 D	1 A 1 F	0 -
5 AREA 11 ACID POND		1 A	0 -	1 A	1 A 1 F	0 -
7A D AREA NORTH LAWN		0 -	0 -	16 A 1 F	0 -	0 -
11A P AREA NORTH		0 -	0 -	4 A	4 A 1 F	0 -
7 D AREA SOUTHEAST DRAINAGE		1 A	0 -	0 -	1 A	0 -
8 D AREA SOUTHWEST DRAINAGE		1 A	0 -	0 -	1 A	0 -
9 D AREA NORTHWEST DRAINAGE		1 A	0 -	0 -	1 A	0 -
10 WATERWORKS NORTH DRAINAGE		1 A	0 -	0 -	1 D 1 G	0 -
11 P AREA SOUTHEAST DRAINAGE		1 A	0 -	0 -	1 A 1 F	0 -
20 D AREA SOUTH		0 -	0 -	0 -	1 A 1 F	0 -
12 AREA 14 LANDFILL		0 -	0 -	1 D	1 A 1 G	0 -
13 AREA 14 CHANGE HOUSE SITE		0 -	0 -	6 A	0 -	0 -
14 AREA 14 SOLVENT STORAGE		2 A	0 -	0 -	2 A 1 F	0 -
15 AREA 7 PLATING POND		1 A	1 M	0 -	1 A	0 -
16 AREA 7 INDUSTRIAL SITE		2 A	0 -	7 A 2 D 1 F 1 G	3 A 1 F	0 -
17 JOB CORPS LANDFILL		2 A	4 G	5 A 2 D 2 G	0 -	0 -
18 AREA 13 LOADING PLATFORM		0 -	0 -	4 A 1 F	0 -	0 -
19 AREA 13 BUNKER 1-3		0 -	0 -	5 A	0 -	0 -

PHASE I SAMPLING & ANALYSIS SUMMARY

(Revised March 10, 1986)

Table 6 pg.2

SITE NO.	SAMPLE TYPE	WATER NO.OF ANAL. SAMPL TYPE	WELL NO.OF ANAL. SAMPL TYPE	SOILS NO.OF ANAL. SAMPL TYPE	SEDIMENTS NO.OF ANAL. SAMPL TYPE	BIOTA NO.OF ANAL. SAMPL TYPE
				1 F		
30 MUNITIONS CONTROL SITE		0 -	1 I	1 D 1 G	0 -	0 -
21 SOUTHEAST CORNER FIELD		0 -	0 -	4 A 1 F	0 -	0 -
22 OLD REFUGE SHOP		1 A	0 -	0 -	1 A 1 F	0 -
24 PEPSI-WEST		1 A	0 -	0 -	1 A 1 F	0 -
25 C.O.CREEK AT MARION LF		3 A	0 -	0 -	2 A 1 D 1 G	0 -
26 C.O.CREEK BELOW MARION STP		2 A	0 -	0 -	2 A	0 -
27 C.O.CREEK BELOW 157 DREDGE		1 A	0 -	0 -	1 D	0 -
28 WATER TOWER LANDFILL		0 -	2 S	11 A 1 D 1 G	0 -	0 -
29 FIRE STATION LANDFILL		0 -	4 S	5 A 2 D 1 G	0 -	0 -
32 AREA 9 LANDFILL		0 -	3 I	1 A 8 B 27 C 9 H	15 A 3 D	0 -
33 AREA 9 BUILDING COMPLEX		0 -	0 -	184 B 4 D	0 -	0 -
35 AREA 9 EAST WATERWAY		0 -	0 -	0 -	1 A 1 F	0 -
34 CRAB ORCHARD LAKE		10 I 5 E	0 -	0 -	8 I 2 H	28 T 2 H
31 REFUGE CONTROL SITE		0 -	1 I	1 D 1 G	0 -	0 -
TOTAL NUMBER OF ANALYSES		36	16	328	71	30
		481				



PHASE I SAMPLING & ANALYSIS SUMMARY

(Revised March 10, 1986)

PHASE I SAMPLING AND ANALYSIS SUMMARY (Revised March 30, 1986)  
ANALYSIS SET

NO. OF ANALYSES	SCREENING					SUB-TOTAL	FULL ANALYSIS				SUB-TOTAL	SELECTED PARAMETERS				TOTAL
	A	B	C	D	E		F	G	H	I		M	N	S	T	
WATER	21	0	0	0	5	26	0	0	0	10	36	0	0	0	0	36
WELL	0	0	0	0	0	0	0	0	0	5	5	1	4	6	0	16
SOILS	72	192	27	15	0	306	6	7	9	0	328	0	0	0	0	328
SEDIMENTS	41	0	0	7	0	48	10	3	2	8	71	0	0	0	0	71
BIODA	0	0	0	0	0	0	0	0	2	0	2	0	0	0	28	30
SUB-TOTAL	134	192	27	22	5	380	16	10	13	23	442	1	4	6	28	481
QA/QC - WATER	1	0	0	0	0	1	0	0	0	3	4	0	1	0	0	5
QA/QC - SOIL	12	31	4	6	0	53	1	2	2	0	58	0	0	2	0	60
QA/QC - SEDIMENT	7	0	0	1	0	8	2	1	1	3	15	0	0	0	0	15
QA/QC - BLANKS	9	0	0	1	0	10	0	2	1	1	14	0	0	0	0	14
QA/QC - TOTAL	29	31	4	8	0	72	3	5	4	7	91	0	1	2	0	94
TOTAL	163	223	31	30	5	452	19	15	17	30	533	1	5	8	28	575

Table 6 pg 3

*1. duplicate of 10/2/87  
2. 10/2/87*

## PHASE II SAMPLING &amp; ANALYSIS SUMMARY

(Revised March 10, 1986)

SITE NO.	SAMPLE TYPE	WATER NO. OF ANAL. SAMPL TYPE	SOILS NO. OF ANAL. SAMPL TYPE	SEDIMENTS NO. OF ANAL. SAMPL TYPE
3	AREA 11 SOUTH LANDFILL	0 -	0 -	0 -
4	AREA 11 NORTH LANDFILL	0 -	0 -	0 -
5	AREA 11 ACID POND	0 -	0 -	0 -
7A	D AREA NORTH LAWN	0 -	0 -	0 -
11A	P AREA NORTH	0 -	0 -	0 -
7	D AREA SOUTHEAST DRAINAGE	0 -	0 -	0 -
8	D AREA SOUTHWEST DRAINAGE	0 -	0 -	0 -
9	D AREA NORTHWEST DRAINAGE	0 -	0 -	0 -
10	WATERWORKS NORTH DRAINAGE	1 J	0 -	5 J
11	P AREA SOUTHEAST DRAINAGE	0 -	0 -	5 K
20	D AREA SOUTH	0 -	0 -	0 -
12	AREA 14 LANDFILL	0 -	0 -	0 -
13	AREA 14 CHANGE HOUSE SITE	0 -	0 -	0 -
14	AREA 14 SOLVENT STORAGE	0 -	0 -	5 L
15	AREA 7 PLATING POND	0 -	0 -	1 N
16	AREA 7 INDUSTRIAL SITE	2 O	0 -	0 -
17	JOB CORPS LANDFILL	2 Q	35 P 12 Q	2 Q
18	AREA 13 LOADING PLATFORM	0 -	0 -	0 -
19	AREA 13 BUNKER 1-3	0 -	0 -	0 -
30	MUNITIONS CONTROL SITE	0 -	0 -	0 -
21	SOUTHEAST CORNER FIELD	0 -	0 -	0 -
22	OLD REFUGE SHOP	0 -	0 -	5 R
24	PEPSI-WEST	0 -	0 -	0 -
25	C.O. CREEK AT MARION LF	0 -	0 -	0 -

also HSL.

This is a supply &amp; study include 51-39.

agreed with 4/35 WP. No. 1 full HSL analyses.

See comments on 4/35 WP. Now about new samples.

See comments on 4/35 WP.

"

"

"

See comments on 4/35 WP.

## PHASE II SAMPLING &amp; ANALYSIS SUMMARY

(Revised March 10, 1986)

SITE NO.	SAMPLE TYPE	WATER NO. OF ANAL. SAMPL TYPE	SOILS NO. OF ANAL. SAMPL TYPE	SEDIMENTS NO. OF ANAL. SAMPL TYPE
26	C.O. CREEK BELOW MARION STP	0 -	0 -	0 -
27	C.O. CREEK BELOW 157 DREDGE	0 -	0 -	0 -
28	WATER TOWER LANDFILL	0 -	0 -	0 - <i>see comments on 7/86 wt.</i>
		<i>well samples</i>		
29	FIRE STATION LANDFILL	0 -	13 S	0 - <i>"</i>
		<i>well samples</i>		
32	AREA 9 LANDFILL	0 -	0 -	59 87 B <i>see comments on 4/86 wt.</i>
33	AREA 9 BUILDING COMPLEX	0 -	151 B	0 - <i>"</i>
35	AREA 9 EAST WATERWAY	0 -	0 -	0 -
34	CRAB ORCHARD LAKE	0 -	0 -	0 - <i>P</i>
31	REFUGE CONTROL SITE	0 -	0 -	0 -
		<i>well samples</i>		
TOTAL NUMBER OF ANALYSES		5	211	80
		296		

NOTE: Well water and lake water and sediment samples were scheduled as part of the PHASE I Sampling program and will be collected and analyzed in PHASE II. Lake fish samples that were collected in PHASE I will also be analyzed in PHASE II.

TABLE 7 pg 3

## PHASE II SAMPLING &amp; ANALYSIS SUMMARY

(Revised March 10, 1986)

## SAMPLING AND ANALYSIS SUMMARY BY SETS

NO. OF ANALYSES	ANALYSIS SET												TOTAL
	B	J	K	L	M	N	O	P	Q	R	S	T	
WATER	0	1	0	0	0	0	2	0	2	0	0	0	5
SOILS	151	0	0	0	0	0	0	35	12	0	13	0	211
SEDIMENTS	57	5	5	5	0	1	0	0	2	5	0	0	80
SUB-TOTAL	208	6	5	5	0	1	2	35	16	5	13	0	296
QA/QC - WATER	0	0	0	0	0	0	0	0	0	0	0	0	0
QA/QC - SOIL	20	0	0	0	0	0	0	0	2	0	2	0	24
QA/QC - SEDIMENT	9	1	1	1	0	0	0	6	0	0	0	0	18
QA/QC - BLANKS	2	1	1	0	0	0	0	1	1	0	0	0	6
QA/QC - TOTAL	31	2	2	1	0	0	0	7	3	0	2	0	48
TOTAL	239	8	7	6	0	1	2	42	19	5	15	0	344

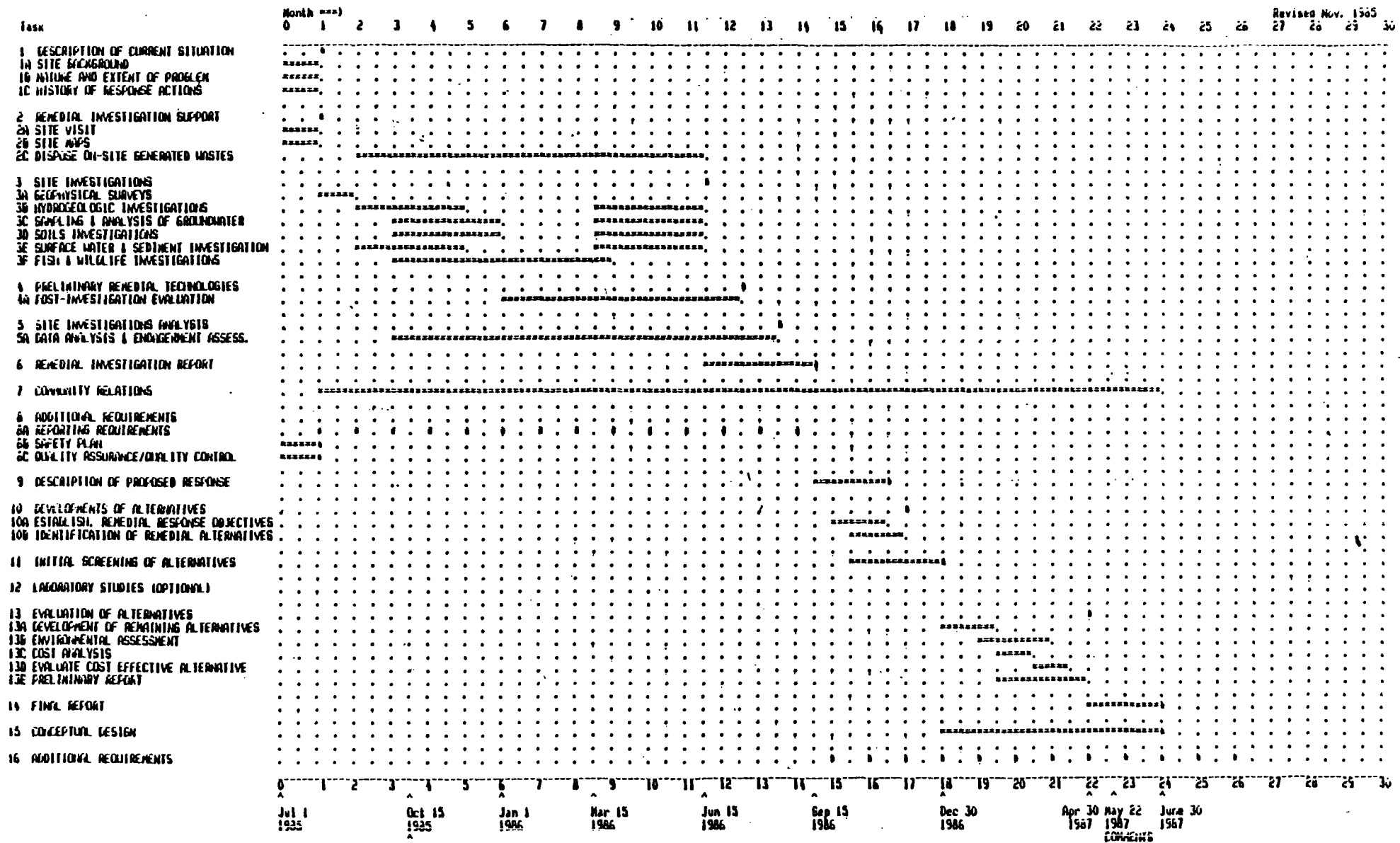
### 1.06 Project Schedule

The proposed project schedule is illustrated in Figure 2. This schedule was developed for planning purposes. Several tasks identified in the Work Plan emphasize uncertainties or contingent items which may be defined at a later date depending on the results of analytical data or engineering assessments. Therefore, schedule modifications may be necessary as these tasks are encountered.

FIGURE 2

## CRAB ORCHARD NATIONAL WILDLIFE REFUGE

## Revised Project Schedule



## SECTION 2 - PROJECT ORGANIZATION AND RESPONSIBILITY

### 2.01 Functional Activities

Table 8 lists the functional activities of this project and the firms responsible for the particular activity.

### 2.02 Project Organization

Table 9 lists the primary contacts for the project. Project technical personnel and quality assurance personnel are indicated in the project organization chart (Figures 3 and 4 respectively). Primary responsibility for project quality review rests in the NWR Resource Contaminants Assessment Coordinator. Independent quality assurance review is provided by the Columbia National Fisheries QA/QC representatives, the refuge manager, and the USEPA On-Scene Coordinator.

### 2.03 Project Manager

← DOI or O&G?

The Project Manager will have primary responsibility for overseeing all facets of the project on a day-to-day basis. Specifically, his duties will include:

- Project scheduling
- Budget control
- Subcontractor performance review
- Review of interim reports
- Responsible for project coordination and communication
- Project deliverables

TABLE 8  
FUNCTIONAL ACTIVITIES

<u>Task/Activity</u>	<u>Responsible Company</u>	<u>Where Performed</u>
<u>Task 1</u> - Description of Current Situation	O'Brien & Gere Engineers, Inc.	Main Office, Syracuse, New York
<u>Task 2</u> - Remedial Investigation Support		
Support - A - Site Visit	O'Brien & Gere Engineers, Inc.	On-Site
B - Site Maps	O'Brien & Gere Engineers, Inc.	Main Office, Syracuse, New York
<u>Task 3</u> - Site Investigations		
A - Geophysical Surveys	O'Brien & Gere Engineers, Inc.	On-Site
B - Hydrogeologic Investigations	O'Brien & Gere Engineers, Inc.	On-Site
- Installation of Monitoring Wells	Professional Service Industries, Inc. with O'Brien & Gere Engineers, Inc. Supervising	On-Site
C - Groundwater: Sampling Analyses	O'Brien & Gere Engineers, Inc. O'Brien & Gere Laboratories, Inc. Environmental Testing & Certification (ETC)	On-Site Laboratory - Syracuse, New York Laboratory - Edison, New York
D - Soil Investigation: Sampling Analyses	O'Brien & Gere Engineers, Inc. O'Brien & Gere Laboratories, Inc.	On-Site Laboratory, Syracuse, New York
E - Surface Water & Sediment Investigation: Sampling Analyses	O'Brien & Gere Engineers, Inc. O'Brien & Gere Laboratories, Inc. Environmental Testing & Certification (ETC)	On-Site Laboratory, Syracuse, New York Laboratory, Edison, New York
F - Biota: Sampling Analyses	O'Brien & Gere Engineers, Inc. O'Brien & Gere Engineers, Inc.	On-Site Laboratory, Syracuse, New York
<u>Task 4</u> - Preliminary Remedial Technologies	O'Brien & Gere Engineers, Inc.	Main Office, Syracuse, New York
<u>Task 5</u> - Site Investigations Analysis	O'Brien & Gere Engineers, Inc.	Main Office, Syracuse, New York
<u>Task 6</u> - Final Report	O'Brien & Gere Engineers, Inc.	Main Office, Syracuse, New York
<u>Task 7</u> - Community Relations	Fish and Wildlife Service	On-Site
<u>Task 8</u> - Additional Requirements	O'Brien & Gere Engineers, Inc.	Main Office, Syracuse, New York



TABLE 9

PRIMARY CONTACTS

<u>Name and Responsibility</u>	<u>Organization and Address</u>	<u>Phone Number</u>
Dr. James Elder Regional Resource Contaminants Assessment Coordinator	U.S. Fish and Wildlife Service Federal Building, Fort Snelling Twin Cities, MN 55111	612/725-3536
Mr. Norrell Wallace Refuge Manager	U.S. Fish and Wildlife Service Crab Orchard National Wildlife Refuge P.O. Box J Carterville, IL 62918	618/997-3344
Dr. Dave Stallings Dr. Jim Petty Quality Control/ Quality Assurance	Columbia National Fisheries Research Laboratory U.S. Fish and Wildlife Service Route 1 Columbia, MO 65201	314/875-5399
Mr. Dick Ruelle Illinois Resource Contaminants Assessment Coordinator	U.S. Fish and Wildlife Service 1830 Second Avenue Rock Island, IL 61201	309/793-5800
Contracting and General Services	U.S. Fish and Wildlife Service Federal Building, Fort Snelling Twin Cities, MN 55111	612/725-3580
Mr. Richard Boice On-Scene Coordinator	U.S. Environmental Protection Agency 230 South Dearborn Street Chicago, IL 64604	312/886-4740
Mr. Bob Cowles Superfund Coordinator	Illinois Environmental Protection Agency 2200 Churchill Road Springfield, IL 62706	217/782-6760
Mr. Joe Stuart Illinois EPA Representative	Illinois Environmental Protection Agency 2209 West Main Marion, IL 62959	618/997-4371
Mr. Mike Carter Illinois Dept. of Conservation Representative	Regional Fish & Wildlife Manager Illinois Dept. of Conservation R.R. 4, Box 68 Benton, IL 62812	Office: 618/435-8138 Home: 618/883-5961

TABLE 9

PRIMARY CONTACTS  
(Continued)

<u>Name and Responsibility</u>	<u>Organization and Address</u>	<u>Phone Number</u>
<i>John Perreconi</i> <del>Ms. Vanessa Musgrave</del> Community Relations	U.S. Environmental Protection Agency 230 South Dearborn Street Chicago, IL 64604	<i>668 5</i> 312/886-6128
Mr. Jim Ross Community Relations	U.S. Fish and Wildlife Service Federal Building, Fort Snelling Twin Cities, MN 55111	612/725-3519
Dr. Robert L. Flentge Illinois Dept. of Public Health Contact	Illinois Dept. of Public Health 525 West Jefferson Springfield, IL 62707	217/785-2439
Mr. Les Frankland Illinois Dept. of Conservation	Illinois Dept. of Conservation 424 Lincoln Tower Plaza Springfield, IL 62706	217/782-6424
Ms. Carol B. Luly Community Relations	Illinois Environmental Protection Agency 2009 Mall Street Collinsville, IL 62234	618/345-6220
Ms. Jean Hutton Office of Soliciter U.S. Department of Interior	U.S. Department of the Interior Room 4354 18th & C Streets, N.W. Washington, D.C. 20240	202/343-5301
Mr. David M. Taliaferro Attorney, U.S. EPA	U.S. Environmental Protection Agency 230 South Dearborn Street Chicago, IL 64604	312/886-6826
Dr. Cornelius B. Murphy, Jr. O'Brien & Gere	O'Brien & Gere Engineers, Inc. P.O. Box 4873 1304 Buckley Road Syracuse, NY 13221	315/451-4700
Mr. John Hanson Beveridge & Diamond	Beveridge & Diamond, P.C. 1333 New Hampshire Ave., N.W. Washington, D.C. 20036	202/828-0285
Ms. Ellen Summer Sangamo Weston, Inc.	Sangamo Weston, Inc. P.O. Box 48400 Atlanta, GA 30362	404/449-9006

FIGURE 3

# PROJECT ORGANIZATION

## REMEDIAL INVESTIGATION/FEASIBILITY STUDY

### CRAB ORCHARD NATIONAL WILDLIFE REFUGE

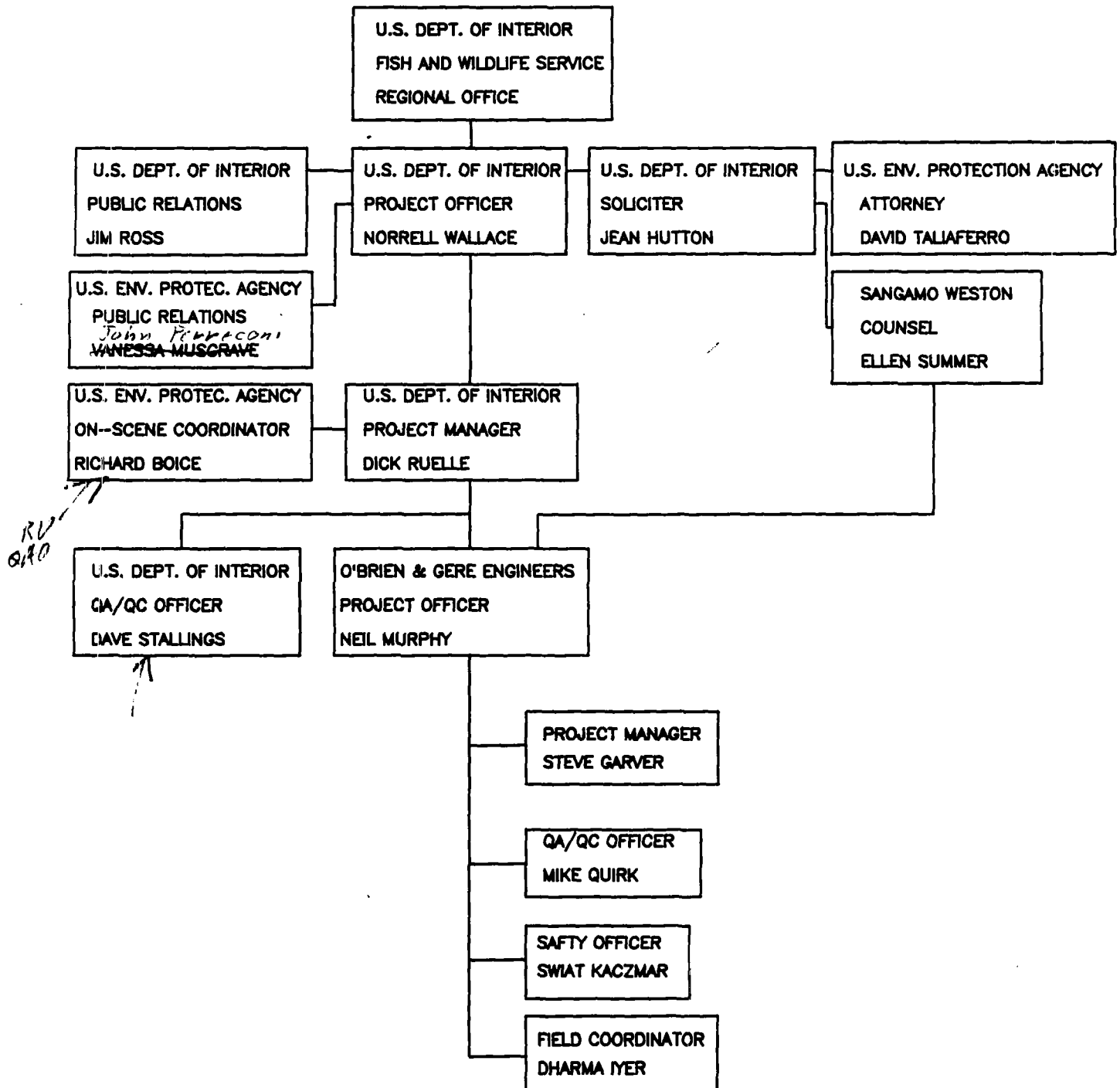
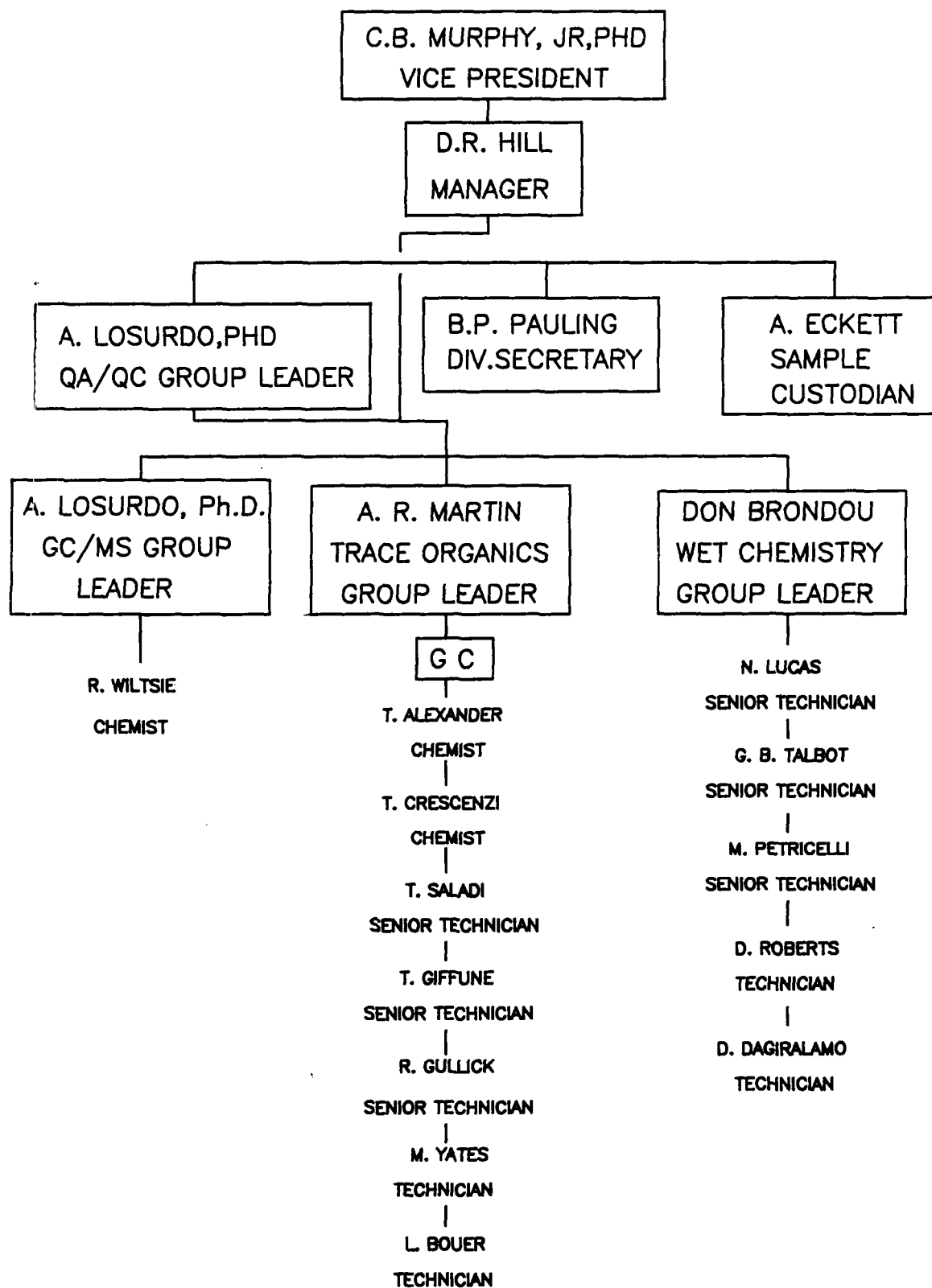


FIGURE 4

## LABORATORY ORGANIZATION CHART



Project Officer?  
Field Coordinator?

Section No: 2  
Revision No: 1  
Date: May 27, 1986  
Page 2 of 2

- Responsible for establishing a project specific record keeping system
- Project close-out

P. Officer  
74 B or DOI

#### 2.04 Quality Assurance Manager

The Quality Assurance (QA) Manager is responsible for the monitoring and supervision of the QA/QC program. The QA Manager reports directly to the Project Manager and his responsibilities include:

- Insure field personnel are both familiar with and adhering to proper sampling procedures, field measurements sample identification and chain-of-custody procedures.
- Contact the laboratory to insure that samples received by them have been properly identified and packaged.
- Maintain a record of performance and system audits and inform the Project Manager of any problems encountered in the analytical procedures.
- The QA Manager in conjunction with the Project and Laboratory Managers will formulate recommendations to correct any deficiency in the analytical protocol or/data.

W/ser. m/  
conduct?

#### 2.05 Assistant Project Managers

The management team for this project will draw upon the technical expertise and experience of a number of different individuals. The project team will consist of multidisciplined personnel with expertise in Aerial Photograph interpretation, hydrogeology, geophysical surveys, chemical characterization, soil science, wet chemistry and risk assessment.

## SECTION 3 - QUALITY ASSURANCE OBJECTIVES

### 3.01 Overall Objectives

The general quality assurance objective for analyzed measurement data is to ensure that environmental monitoring data of known and acceptable quality are provided.

*See this figure?*

For this project, the ~~specific~~ objectives for measurement data in terms of precision, accuracy and compatibility are the same as the objectives established for the Statement of Work for the U.S. EPA Contract Laboratory Program (CLP), viz.: The purpose of the QA/QC program....is the definition of procedures for the evaluation and documentation of subsampling, analytical methodologies, and the reduction and reporting of data. The objectives is to provide a uniform basis for subsampling, sample handling, instrument condition, methods control, performance evaluation, and analytical data generation and reporting." This QAPP for sampling, analysis and data handling is consistent with the requirement set forth by the U.S. Fish and Wildlife Service, as well as all State and Federal EPA requirements.

### 3.02 Field QC Objectives and Procedures

Field functions such as; magnetometer and electromagnetic terrain conductivity services are activities which do not include sample collection, but involve measurements where quality assurance concerns are appropriate. The primary objective in activities such as these is to obtain reproducible measurements consistent with their intended use.

The methods employed in conducting these magnetometer and electromagnetic terrain conductivity surveys are included as Attachments 3 and 4.

The objective of sampling procedures is to obtain samples that represent the environmental matrix being investigated. Trace levels of contaminants from external sources will be eliminated through the use of good sampling techniques and proper selection of sampling equipment.

A detailed description of sampling procedures is presented in the Site Sampling Plans for Phase I (December 1985) and Phase II (April 1986). Source material used in developing the sampling plan included the following:

Technical Support Documents

- Samplers and Sampling Procedures for Hazardous Waste Streams (EPA-600/2-80-180)
- Test Methods for Evaluating Solid Wastes (EPA SW 846-1980)
- User's Guide to the EPA Contract Laboratory Program
- EPA Technique Monographs
  - 15--Purposes and Objectives of Sampling
  - 16--Water Sampling Methods
  - 17--Soil and Sediment Sampling Methods
  - 18--Sampling of Biological Specimens
  - 19--Methods of Collecting Concentrated (Hazardous) Samples
  - 20--Container Opening Techniques
  - 22--Sample Handling, Packaging, and Shipping

Procedures

The Site Sampling Plans include the following protocols and documentation.

- Number of locations to be sampled
- Sampling procedures to be used at the site
- Tests to be completed at each sampling location
- Sampling equipment required at the site
- Sample containers required at the site
- Preservation methods to be used at the site for various types of samples
- Reagents, etc., required at the site for sample preservation
- Shipping containers required at the site
- Chain-of-custody procedures to be used at the site
- Shipping methods and destinations, marking instructions, special labels, etc.

### 3.03 Field QC Audits

Blanks and duplicate samples will be collected as part of our QA/QC program. Blanks are employed to ensure that neither glassware nor procedural contamination has occurred. Additionally, they are utilized to evaluate ambient site conditions which may cause sample contamination. If positive interferences occur, the Quality Assurance Manager (QAM) will recommend to the Project Manager that sample collecting and handling procedures be technically reviewed to eliminate such sample contamination.



Duplicate samples are treated throughout as two unique samples. The results of duplicate analyses provide information on the overall precision of both the sampling and analytical programs.

The number of duplicate and spikes samples for Phase I and II are summarized in Tables 6 and 7 respectively.

### 3.04 Accuracy, Sensitivity and Precision of Analysis

All samples collected, (soil, water and sediments) will be analyzed using the Contract Laboratory Program (CLP). Parameters, method detection limits audit, frequency and central limits are shown in Table 10.

*This is not true!*

*This must be clearly specified for each sample. It is do not know whether had w/ 1 (A Por 239.1,*

## SECTION 4 - SAMPLING PROCEDURES

### Objective

The objective of this Sites Sampling Plan (SSP) is to document the sampling locations, procedures and practices that will be used in the Remedial Investigation sampling program to be conducted at Crab Orchard National Wildlife.

It is anticipated that the sampling and analysis program at Crab Orchard National Wildlife Refuge will be accomplished in two phases.

Phase 1 will be the basis used to determine if a potential problem (s) exists on a specific site and to characterize the range of chemical compounds which contribute to the problem. Phase II will be employed to define the extent of contamination (both vertically and laterally) of any site identified during Phase 1 as a area of concern. The information obtained during Phase II will be used in evaluating the remedial options.

In general, the analytical effort associated with Phase II will be less than that of Phase I, because the results of the initial effort will assist in diminishing the total number of sites and reducing both organic and inorganic constituents of concern.

### Types of Samples

Various matrices will be sampled and analyzed as part of the Remedial Investigation. These include the following:

1. Waters: including groundwaters, surface streams, raw and finished water supplies, pond waters and waters from Crab Orchard Lake.
2. Sediments: from streams, ponds and Crab Orchard Lake.
3. Soils: including soils potentially affected by surface spillage and fill material from sites of past disposal activity.
4. Air: as part of the site safety program.
5. Biota: including fish, turtles and crayfish.

For the most part, all samples will be obtained as single grab samples. No time-composited samples are contemplated at this time. However, at many sites, areal soil composites will be prepared. Areal composites are used as a screening device to allow initial assessments of broad areas for a range of contaminants. Compositing procedures are discussed below.

#### Compositing Procedures

Areal composites of water samples (along stretches of streams, surfaces of ponds or depth composites in Crab Orchard Lake) will be prepared by combining equal volumes of grab samples at each location. Individual grab samples for volatile organic analyses will be retained and labelled in individual headspace-free vials for compositing by the laboratory.

Areal composites of soil samples will be prepared either in the field or in the laboratory after refrigerating individual grabs to 0 to 4°C. This will minimize loss of volatile materials. Where soils are obtained in Lexan cores, these will be capped and refrigerated prior to compositing.

*I have not being done on Phase II eliminate*

## General Sampling Locations and Numbers

### Sample Locations

*eliminate*

Sampling locations were determined in the field during a site reconnaissance visit on March 26-28, 1985. They are presented in the Site Sampling Plan (Dec. 1985). A log book listing the various samples to be collected will be prepared for use on-site. The log book will also contain the type of sample and analytical matrix for each of the samples to be collected. Pre-printed peel-off labels will be included in the log book for tagging the various containers to be used for sample collection. The sample team leader will be responsible for determining the exact sampling location and recording the location in the field sampling notebook. The location will be described in the log book with a sketch that includes distances from numbered field reconnaissance stakes and other landmarks. The rationale of selecting a sampling location will also be included. All sampling locations will be photographed.

### Sample Numbering System

A sample numbering system will be used to identify each sample taken during the remedial investigation sampling program. This numbering system will provide a tracking procedure to allow retrieval of information regarding a particular sample and to assure that each sample is uniquely numbered. A listing of the sample identification numbers will be maintained by the sample team leader.

## Sampling Equipment and Sampling Procedures

### Soil Sampling

*sample?*  
↓  
Soil samples will be collected from identified spots around the Refuge and during the installation of additional groundwater monitoring wells. Samples will be collected in general accordance with the split spoon sampling procedure (ASTM D1586-67), using 2-inch OD split spoon samplers.

*even for surface soils?*

### Groundwater Studies and Sampling

*reference method when?*  
Aquifer slug recovery tests will be conducted in all additional monitoring wells to obtain in situ estimates of hydraulic conductivity. A minimum of two test runs should be made at each test well.

*reference?*  
Properly decontaminated equipment will be used in sampling all groundwater monitoring wells. See the Decontamination protocols in Attachment 3 of the QAPP. Before samples are taken, each well will be purged until there is a constant conductivity, (usually about 5 to 10 well volumes). After the well has recovered, samples for inorganic and organic (excluding volatiles) analysis can be collected using a peristaltic pump or hand bailer. Samples to be analyzed for volatile organics will be collected by bailing. Teflon tubing will be used for the suction and discharge lines for peristaltic pumps. Hand bailers will be constructed of stainless steel or Teflon.

*not useful.*

### Waste Sampling

The Area 9 Landfill is the only site of the Refuge where waste materials are being sampled. All other sites represent sampling of matrices potentially affected by dispensed contaminants. There are special safety concerns posed by the sampling of waste materials at Area 9 because of the possible presence of explosives residues or even undetonated cartridges. Similar concerns exist at other sampling sites, but sampling elsewhere is limited to within 1 foot from the surface. Soil borings at Area 9 will employ split spoon sampling procedures. Drilling personnel will be required to be removed at least 100 ft. from the drill rig during advancement of the augers. This is further discussed in the SHSP.

### Field Blanks

Field Blanks for sediment and soil samples will consist of analytical grade diatomaceous earth. For water samples, ultrapure distilled/deionized water will be used. The field blank sample will be placed into the appropriate sampling equipment, removed from the equipment, and then placed into sampling containers.

### Duplicate Samples

Duplicate samples are defined as two distinct samples taken from the same location at similar times using identical sampling equipment that has been decontaminated in a similar manner.

However, duplicate samples of soil cores will consist of a given core homogenized, divided equally and submitted for analysis as two distinct samples.

### Split Samples

A number of samples will be split with a representative of the FWS for analysis. Split samples are defined as one distinct sample that is divided equally and sent to two different laboratories for analysis. Soils will be field homogenized in a clean aluminum pan prior to splitting. Water sample splits will be duplicates.

### General Decontamination Procedures

Decontamination of personal gear (boots, gloves, and waders), sample jars and sampling equipment will be as follows (see also attached materials to the SHSP):

1. Wash personal gear or sample containers in a bucket or tub filled between 50 and 75 percent with a trisodium phosphate (TSP) solution (2 lbs of TSP per 10 gallons of clean water). Completely brush the entire exterior surface of the article undergoing decontamination. If PCB's are expected to be present, add 4 lbs of sodium bicarbonate per 10 gallons of water to the washing solution.
2. Rinse personal gear or sample containers in a bucket or tub filled between 50 and 75 percent with clean water. Completely brush the entire exterior surface of the article undergoing decontamination.

*Special procedures req. including taking in water for organic HSL's, Nitric acid rinse for metals, use CIP protocols*

*Each jar should be washed in separate batches*

3. Dispose of all wash and rinse water in a properly marked and sealed container. All such containers of wastewater will be stored in a secure area on-site and properly disposed of during the remedial action phase.

### Sampling Equipment

1. Wash sampling equipment in a bucket or tub filled between 50 and 75 percent with a TSP solution (2 lbs of TSP per 10 gallons of clean water). Completely brush the entire exterior surface of the article undergoing decontamination. Wash interior wetted surfaces as required. If PCB's are expected to be present, add 4 lbs of sodium bicarbonate to the washing solution. Drilling equipment, augers and split spoon samplers can be decontaminated by steam cleaning using clean water.
2. Rinse only heavily contaminated sampling equipment in a bucket or tub filled between 50 and 75 percent with a 20 percent solution of acetone and water. Completely brush the entire exterior surface of the article undergoing decontamination. Rinse interior wetted surfaces as required. *If PCB's are present, the first rinse should be carried out with a hexane solution.*
3. Following step 2 above, rinse all sampling equipment in a bucket or tub filled between 50 and 75 percent with distilled water. Completely brush the entire exterior surface of the article undergoing decontamination. Rinse interior wetted surfaces as required.



4. Collect all wash and rinse water in a properly marked and sealed container. Wash and rinse water will be analyzed relative to its hazardous waste characteristics and disposed of in accordance with all applicable state and federal regulations. Drilling soils and water as well as discarded protective clothing will be treated similarly.

#### Screening Procedures

It is probable that not all soil samples will have significant concentrations of contaminants. To reduce analytical costs, a field screening procedure may be used in Phase II of the RI to reduce the number of soil samples sent for complete laboratory analysis.

While constituents used for screening may not be the only contaminants present, they may be used as an indicator of contamination. If they are present in a sample in concentrations exceeding the positive response criteria established in the Work Plan, the interpretation that other contaminants may also be present will be made and the sample will be sent to the laboratory for analysis of constituents established in the Work Plan (June 1985).

#### Documentation

##### Site Location Procedure

Following sampling location identification, a wood stake (approximately 2" X 2" X 24") will be driven into the ground, allowing approximately 8 to 10 inches of the stake to remain visible above ground. The top portion of the stake will be painted orange and labeled for identification. The label will contain sample

number and sample type. The location of each stake will be recorded. Sample locations will eventually be surveyed and tied into the site grid system.

### Photographs

Photographs (35mm, color slides) will be taken to illustrate sampling locations. Photographs will show the surrounding area and reference objects which help to locate sampling sites. The picture number and roll number (if more than one roll of film is used) will be logged in the field notebook to identify which sampling site is depicted in the photograph. The film roll number will be identified by taking a photograph of an informational sign on the first frame of the roll. This sign would have the job and film roll number written on it to identify the pictures contained on the roll.

### Field Notebooks

Field notebooks will provide the means of recording data on collecting activities performed at a site. As such, entries will be described in as much detail as possible so that anyone going to the site could reconstruct a particular situation without reliance on memory.

Field notebooks will be bound. Notebooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each notebook will be identified by the project-specific document number.

The cover of each notebook will contain:

Person or Organization to whom the book is assigned.

Book Number

Project Name

Start Date

End Date

Entries into the notebook will contain a variety of information.

At the beginning of each entry, the date, start time, weather, all field personnel present, level of personal protection being used onsite, and the signature of the person making the entry will be entered. The names of visitors to the site, all field sampling team personnel and the purpose of their visit will be recorded in the field notebook.

All measurements made and samples collected will be recorded. All entries will be made in ink with no erasures allowed. If an incorrect entry is made, it will be crossed out with a single strike mark. Wherever a sample is collected or a measurement is made, a detailed description of the location of the station, which includes compass and distance measurements, shall be recorded. The film roll number and number of photographs taken of the station will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the procedures documented in this plan. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers.

In addition, the cooler number into which the sample is placed in the field will be recorded. Sample numbers will be assigned prior to going onsite. Duplicates, which will receive an entirely separate sample number, will be noted under sample description. Significant field notebook entries (samples collected, significant observations) shall be countersigned by another member of the project team.

#### Control of Contaminated Sampling Materials

Disposable sampling and safety equipment and excess samples may be generated during sampling operations. These materials will be placed in 55-gallon drums (separate drums for solids, decontamination liquids, debris, and disposable equipment). Decontamination liquids should also be separated based on those containing solvents (acetone, hexane, etc.) and those containing only detergents (TSP, etc.). The drums will be sealed, labelled and properly stored in a secure area for proper, legal disposal during the remedial action phase. Bailed well water and contaminated drilling spoils will be drummed for proper storage in a secure area.

#### Sample Control

Serialized sample tags will be used to label each sample for analysis. Chain-of-custody records will be completed for all samples according to EPA requirements and procedures set forth in

NEIC Policies and Procedures EPA-330-19-78-001R. Custody seals will be placed on all shipping coolers containing samples.

#### Sample Containers and Sample Preservation

Required sample containers, filling instructions and preservation procedures are listed in Table 1 of Attachment 1 of this SSP. The collected samples will be kept out of direct sunlight and, after decontamination and labeling, will be placed in coolers for shipment to the analytical laboratory.

#### Sample Shipping

Samples will be packed and labelled according to DOT regulations and protocols appearing in Attachment 1 of this SSP. Samples will be shipped via a 24 hour delivery service to the analytical laboratory so that the samples can be extracted within allowable time limits (See QAPP).

## SECTION 5 - SAMPLE CUSTODY

### 5.01 General

Sample custody procedures for this project will be in strict conformance with the procedures detailed in NEIC Policies and Procedures (EPA-3309-78-001-R). These procedures were established to comply with EPA requirements for sample control. They are documented in Attachment 4 to this QAPP.

All samples collected for analysis will be taken by chemists, physical science technicians, or other qualified personnel designated by O'Brien & Gere with specific instructions from the Project Manager. The FWS will take duplicate samples at a ratio of 1:10 for QA/QC purposes. All samples for residue analysis will be placed in the custody of the analytical chemist responsible for the analysis. The sample information will be recorded on the same report sheets if analyzed immediately. Stored sample (including archive portions) will be catalogued and stored may be audited by the QA Officer. Subsequent to approval of the conceptual design (Task 15), these archived samples will be returned to CONWR for disposal consistent with the remedial action plan.

### 5.02 Chain of Custody Procedures

The consequences of an uncontrolled hazardous waste site investigation are difficult to predict. There is a possibility that several years after the RI/FS is complete there will be litigation. For that reason, it is imperative that an accurate record be maintained and documented of sample collection, transport, analysis and disposal.

Therefore, chain of custody procedures are instituted and followed throughout the study.

Chain of custody procedures include field custody, laboratory custody, and evidence files. Samples are physical evidence and should be handled according to procedural safeguards. The project coordinator must be prepared to produce documentation that traces the samples from the field to the laboratory and through the analysis. The National Enforcement Investigation Center (NEIC) of the U.S. EPA defines custody of evidence in the following ways:

- In actual physical possession
- In view after being in physical possession
- In a locked repository
- In a secure, restricted area

Chain of custody records begin in the field when sample collection has been completed. See Figure "Chain of Custody Form" for a typical arrangement of the paper samplers use to complete their field logs. On that form, they note meteorological data, equipment employed during collection, evacuation techniques and any calculations, physical characteristics of samples, date, time of day and location, any abnormalities during sampling.

The sampler completes the custody form, packages the samples including the custody form, and seals the package with evidence tape. Shipment may be made by commercial vendors, and their policy is to document the transfer of the package within their organization. Therefore, when the sample arrives at the laboratory, the sample

custodian signs the vendors air bill or bill of lading. The sample custodian's duties and responsibilities upon sample receipt are:

- Document receipt of samples.
- Inspect sample shipping containers for presence or absence of custody seals, locks, evidence tape, container integrity.
- Record condition of shipping and sample containers in logs.
- Sign appropriate forms or documents.
- Verify and record agreement or disagreement of information on sample documents. If there is discrepancy, record the problem and notify the project officer.
- Label sample with laboratory sample number.
- Place samples in storage, including secure storage, if appropriate.

The hand-to-hand custody of samples in the laboratory is maintained through preparation and analysis. The analyst is required to log samples into and from secure storage as the analysis proceeds. Samples are returned to secure storage at the close of business. Log sheets incorporate options for multiple entries, because several people handle the samples throughout the analytical scheme. See Figure, "Chain of Custody Form for Analysis."

The laboratory records may also be used as evidence in enforcement proceedings, therefore care must be exercised to properly complete, date and sign items needed to generate data. Copies of the following items are stored:

- Documentation of the preparation and analysis of samples, including copies of the analyst's notebooks.



- Bench sheets, graphs, computer printouts, chromatographic outputs, mass spectral outputs.
- Copies of all QA/QC data.
- Instrument logs showing date, time and analyst.
- Analytical tracking forms which record date, time, and analyst for each step of sample preparation and analysis.

Upon completion of analysis, the project officer or his assignee should commence assimilating all the field and laboratory notes. It is they who generate the evidence file for the project. The package is arranged in chronological order for ease of review. When all the information is gathered, the package is inventoried, numbered and stored for future reference. The document inventory list is illustrated in the following Table:

DOCUMENT CONTROL NUMBER	TYPE	NO. OF PAGES
1111-1	Project file inventory sheets	1
1111-2	Field notes	30
1111-3	Chain-of-custody records	7
1111-4	Shipping manifests	27
1111-5	Sample log-in sheets	40
1111-6	Sample control records	40
1111-7	Sample tickets	500
1111-8	Sample traffic reports	127
1111-9	Analytical traffic reports	127
1111-10	Analytical data summary	10
1111-11	Sample #2	20
1111-12	Sample #3	20
1111-62	Sample #50	20
1111-63	Lab notebook pages	37
1111-64	Bench sheets	50
1111-65	Instrument log pages	13
1111-66	Copies of mass spectral data, graphs, chromatograms	43
1111-67	Related correspondence	4

## SECTION 6 - EQUIPMENT CALIBRATION

### 6.01 Calibration Procedures

#### Equipment Calibration, References and Frequency

All field equipment used during this project will be calibrated and operated in accordance with manufacturer's instructions. Any field equipment used during this project that is not covered by the investigator's standard operating procedures will have a specific calibration and operation instruction sheet prepared for it.

#### A. General

Standards may be generally grouped into two classifications: primary and secondary. Primary standards include USP and NE drugs, NBS and ASTM materials, and certain designated EPA reference materials. All other standards are to be considered secondary.

#### B. Testing

1. Primary: No testing is necessary. Do not use if there is any physical indication of contamination or decomposition (i.e. partially discolored, etc.).
2. Secondary: Examine when first received either by comparison to an existing primary, or comparing known physical properties to literature values. The less stable standards will be rechecked at appropriate intervals, usually six months to one year.

C. Records

1. A records book will be maintained for each grouping of standards (i.e. pesticides, metals, etc.)
2. The record kept for each standard will include:
  - a. Name and date received
  - b. Source
  - c. Code or lot number
  - d. Purity
  - e. Testing data including all raw work and calculations
  - f. Special storage requirements
  - g. Storage location
3. These records will be checked periodically as part of the Laboratory Controls Review.

Equipment

A. General

1. Each major piece of analytical laboratory instrumentation used on this project is documented and on file with the analytical laboratory.
2. A form is prepared for each new purchase and old forms will be discarded when the instrument is replaced.

B. Testing

1. Each form details both preventative maintenance activities and the required QA testing and monitoring.
2. In the event the instrument does not perform within the limits specified on the monitoring form, the Laboratory Manager will be notified and a decision made as to what action to take.
3. If repair is deemed necessary, an "out of order" sign will be placed in the instrument until repairs are effected.

6.02 Calibration Records

A bound notebook will be kept with each instrument, requiring calibration, to record all activities associated with a maintained, QA monitoring and repairs program. Additionally, these records will be checked during periodic equipment review.

## SECTION 7 - ANALYTICAL PROCEDURES

### 7.01 Laboratory Analytical Procedures

The analysis and methods detection limits for standard CLP procedures are given in Table 10 (see Section 3.04).

The methods associated with:

- Volatile organics in water
  - Volatile organic screens in soil and sediment
  - Semi-volatile FID scans of water and soil samples
  - Organo chlorine pesticides and PCBs in water and soil samples
- are attached to the end of this section.

Additionally, the laboratory analyzed a variety of matrices for a number of different environmental constituents of concern. Therefore, several documents are referenced which include the procedures employed. The following list itemizes the most widely used documents.

1. Standard Methods for the Examination of Water and Wastewater
2. Methods for Chemical Analysis of Water and Wastewater
3. ASTM Annual Book of Standards
4. Code of Federal Regulations
5. NIOSH Manual of Analytical Methods
6. Test Methods for Evaluating Soil Waste, Physical/Chemical Methods

When analyzing samples by the above standardized methods, the accuracy or precision of the data generated by the laboratory is determined through analysis of replicates, spiked samples, synthetic

Table 10 Page 1 of 36  
ANALYTICAL METHOD: WATER  
ACID EXTRACTABLES

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
	(GC)	ppb	(GC/MS)	ppb	(CLP)	ppb			
2,4,6-trichlorophenol	604	10	625	10	WA 85-177	10	SEE BELOW	SEE BELOW	SEE BELOW
2,4-dichlorophenol	604	10	625	10	WA 85-177	10	"	"	"
2,4-dimethylphenol	604	10	625	10	WA 85-177	10	"	"	"
2,4-dinitrophenol	604	50	625	50	WA 85-177	50	"	"	"
2-chlorophenol	604	10	625	10	WA 85-177	10	"	"	"
2-fluorophenol	604	10	625	10	WA 85-177	10	"	"	"
2-methyl-4,6-dinitrophenol	604	20	625	20	WA 85-177	50	"	"	"
2-nitrophenol	604	20	625	20	WA 85-177	10	"	"	"
4-chloro-3-methylphenol	604	10	625	10	WA 85-177	10	"	"	"
4-nitrophenol	604	50	625	50	WA 85-177	50	"	"	"
Acid Extractable Screen							"	"	"
pentachlorophenol	604	50	625	50	WA 85-177	50	"	"	"
pentafluorophenol	604	20	625	20	WA 85-177	50	"	"	"
phenol	604	10	625	10	WA 85-177	10	"	"	"

Table 10 Page 2 of 36  
ANALYTICAL METHOD: WATER  
BASE/NEUTRALS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
	(GC)	ppb	(GC/MS)	ppb	(CLP)	ppb			
							SEE BELOW	SEE BELOW	SEE BELOW
1,2,4-trichlorobenzene	612	10	625	10	WA 85-177	10	"	"	"
1,2-dichlorobenzene	602	10	625	10	WA 85-177	10	"	"	"
1,2-diphenylhydrazine	-	-	625	20	WA 85-177	10	"	"	"
1,3-dichlorobenzene	602	10	625	10	WA 85-177	10	"	"	"
1,4-dichlorobenzene	602	10	625	10	WA 85-177	10	"	"	"
2,4-dinitrotoluene	609	10	625	10	WA 85-177	10	"	"	"
2,6-dinitrotoluene	609	10	625	10	WA 85-177	10	"	"	"
2-chloronaphthalene	612	10	625	10	WA 85-177	10	"	"	"
3,3-dichlorobenzidine	605	50	625	50	WA 85-177	10	"	"	"
4-bromophenyl phenyl ether	611	10	625	10	WA 85-177	10	"	"	"
4-chlorophenyl phenyl ether	611	10	625	10	WA 85-177	10	"	"	"
acenaphthalene	610	10	625	10	WA 85-177	10	"	"	"
acenaphthene	610	10	625	10	WA 85-177	10	"	"	"
anthracene	610	10	625	10	WA 85-177	10	"	"	"
Base/Neutral Screen							"	"	"
benzidine	605	50	625	50	WA 85-177	10	"	"	"
benzo(a)anthracene	610	10	625	10	WA 85-177	10	"	"	"
benzo(a)pyrene	610	10	625	10	WA 85-177	10	"	"	"
benzo(b)fluoranthene	610	10	625	10	WA 85-177	10	"	"	"
benzo(g,h,i)perylene	610	20	625	20	WA 85-177	10	"	"	"
benzo(k)fluoranthene	610	10	625	10	WA 85-177	10	"	"	"
bis(2-chloroethoxy) methane	611	10	625	10	WA 85-177	10	"	"	"
bis(2-chloroethyl) ether	611	10	625	10	WA 85-177	10	"	"	"
bis(2-chloroisopropyl) ether	611	10	625	10	WA 85-177	10	"	"	"
bis(2-ethylhexyl)phthalate	606	10	625	10	WA 85-177	10	"	"	"
butyl benzyl phthalate	606	10	625	10	WA 85-177	10	"	"	"
chrysene	610	10	625	10	WA 85-177	10	"	"	"
di-n-butylphthalate	606	10	625	10	WA 85-177	10	"	"	"
di-n-octyl phthalate	606	10	625	10	WA 85-177	10	"	"	"
dibenzo(a,n)anthracene	610	20	625	20	WA 85-177	10	"	"	"

Table 10 Page 3 of 36  
ANALYTICAL METHOD: WATER  
BASI /NEUTRALS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
diethyl phthalate	606	10	625	10	WA 85-177	10	"	"	"
dimethyl phthalate	606	10	625	10	WA 85-177	10	"	"	"
fluoranthene	610	10	625	10	WA 85-177	10	"	"	"
fluorene	610	10	625	10	WA 85-177	10	"	"	"
hexachlorobenzene	612	10	625	10	WA 85-177	10	"	"	"
hexachlorobutadiene	612	10	625	10	WA 85-177	10	"	"	"
hexachlorocyclopentadiene	612	10	625	10	WA 85-177	10	"	"	"
hexachloroethane	612	10	625	10	WA 85-177	10	"	"	"
indeno(1,2,3-c,d)pyrene	610	20	625	20	WA 85-177	10	"	"	"
isophorone	603	10	625	10	WA 85-177	10	"	"	"
N-nitrosodi-n-propylamine	607	20	625	20	WA 85-177	10	"	"	"
N-nitrosodimethylamine	607	50	625	50	WA 85-177	10	"	"	"
N-nitrosodiphenylamine	607	20	625	20	WA 85-177	10	"	"	"
naphthalene	610	10	625	10	WA 85-177	10	"	"	"
nitrobenzene	603	10	625	10	WA 85-177	10	"	"	"
phenanthrene	610	10	625	10	WA 85-177	10	"	"	"
pyrene	610	10	625	10	WA 85-177	10	"	"	"



Table 10 Page 4 of 36  
ANALYTICAL METHOD: WATER  
DIOXINS/FURANS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
		ppb		ppt		ppb			
							SEE BELOW	SEE BELOW	SEE BELOW
tetra-CDD			SEE NOTE 9	2			"	"	"
tetra-CDF			SEE NOTE 9	2			"	"	"
penta-CDD			SEE NOTE 9	2			"	"	"
penta-CDF			SEE NOTE 9	2			"	"	"
hexa-CDD			SEE NOTE 9	2			"	"	"
hexa-CDF			SEE NOTE 9	2			"	"	"
hepta-CDD			SEE NOTE 9	20			"	"	"
hepta-CDF			SEE NOTE 9	20			"	"	"
octa-CDD			SEE NOTE 9	20			"	"	"
octa-CDF			SEE NOTE 9	200			"	"	"

NOTE 9 - Determination of Parts-per-Trillion levels of polychlorinated Dibenzofuran and dioxins in environmental samples, Smith L.M., Johnson J.C., Analytic Chemistry 1984, 56, 1830-1842, September 1984.

Table 10 Page 5 of 36  
ANALYTICAL METHOD: WATER  
PESTICIDES/PCBs

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
	(GC)	ppb	(GC/MS)	ppb	(CLP)	ppb			
4,4'-DDD	600	0.1	625	10	WA 85-177	10	SEE BELOW	SEE BELOW	SEE BELOW
4,4'-DDE	600	0.1	625	10	WA 85-177	10	"	"	"
4,4'-DDT	600	0.1	625	10	WA 85-177	10	"	"	"
aldrin	600	.05	625	10	WA 85-177	10	"	"	"
Aroclor 1016	600	.5	625	10	WA 85-177	10	"	"	"
Aroclor 1221	600	.5	625	10	WA 85-177	10	"	"	"
Aroclor 1232	600	.5	625	10	WA 85-177	10	"	"	"
Aroclor 1242	600	.5	625	10	WA 85-177	10	"	"	"
Aroclor 1248	600	.5	625	10	WA 85-177	10	"	"	"
Aroclor 1254	600	1	625	10	WA 85-177	10	"	"	"
Aroclor 1260	600	1	625	10	WA 85-177	10	"	"	"
chlordane	600	.5	625	10	WA 85-177	10	"	"	"
dielzin	600	1	625	10	WA 85-177	10	"	"	"
endosulfan I	600	.05	625	10	WA 85-177	10	"	"	"
endosulfan II	600	1	625	10	WA 85-177	10	"	"	"
endosulfan sulfate	600	1	625	10	WA 85-177	10	"	"	"
endrin	600	1	625	10	WA 85-177	10	"	"	"
endrin aldehyde	600	1	625	10	WA 85-177	10	"	"	"
endrin ketone	600	1	625	10	WA 85-177	10	"	"	"
heptachlor	600	.05	625	10	WA 85-177	10	"	"	"
heptachlor epoxide	600	.05	625	10	WA 85-177	10	"	"	"
methoxychlor	600	.5	625	10	WA 85-177	10	"	"	"
toxaphene	600	1	625	10	WA 85-177	10	"	"	"
β-BHC	600	.05	625	10	WA 85-177	10	"	"	"
γ-BHC (lindane)	600	.05	625	10	WA 85-177	10	"	"	"
α-BHC	600	.05	625	10	WA 85-177	10	"	"	"

Table 10 Page 6 of 36  
ANALYTICAL METHOD: WATER  
VOLATILES

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
	(GC)	ppb	(GC/MS)	ppb	(CLP)	ppb			
							SEE BELOW	SEE BELOW	SEE BELOW
1,1,1-trichloroethane	601	1	624	10	WA 85-177	10	"	"	"
1,1,2,2-tetrachloroethane	601	1	624	10	WA 85-177	10	"	"	"
1,1,2-trichloroethane	601	1	624	10	WA 85-177	10	"	"	"
1,1-dichloroethane	601	1	624	10	WA 85-177	10	"	"	"
1,1-dichloroethene	601	1	624	10	WA 85-177	10	"	"	"
1,2-dichloropropane	601	1	624	10	WA 85-177	10	"	"	"
2-bromo-1-chloropropane	601	1	624	10	WA 85-177	10	"	"	"
2-butanone	602	10	624	10	WA 85-177	10	"	"	"
2-chloroethylvinyl ether	601	10	624	10	WA 85-177	10	"	"	"
2-hexanone	602	10	624	10	WA 85-177	10	"	"	"
4-methyl-2-pentanone	602	10	624	10	WA 85-177	10	"	"	"
acetone	601	10	624	50	WA 85-177	10	"	"	"
benzene	602	1	624	10	WA 85-177	10	"	"	"
bromochloromethane	601	1	624	10	WA 85-177	10	"	"	"
bromodichloromethane	601	1	624	10	WA 85-177	10	"	"	"
bromoform	601	10	624	10	WA 85-177	10	"	"	"
bromomethane	601	10	624	50	WA 85-177	10	"	"	"
c-1,3-dichloropropene	601	1	624	10	WA 85-177	10	"	"	"
carbon tetrachloride	601	1	624	10	WA 85-177	10	"	"	"
chlorobenzene	602	1	624	10	WA 85-177	10	"	"	"
chloroethane	601	10	624	50	WA 85-177	10	"	"	"
chloroform	601	1	624	10	WA 85-177	10	"	"	"
chloromethane	601	10	624	50	WA 85-177	10	"	"	"
dibromochloromethane	601	1	624	10	WA 85-177	10	"	"	"
dichlorodifluoromethane	601	1	624	10	WA 85-177	10	"	"	"
ethyl benzene	602	1	624	10	WA 85-177	10	"	"	"
methylene chloride	601	1	624	10	WA 85-177	10	"	"	"
t-1,1-trichloroethene	601	1	624	10	WA 85-177	10	"	"	"
t-1,3-dichloropropene	601	1	624	10	WA 85-177	10	"	"	"
tetrachloroethene	601	1	624	10	WA 85-177	10	"	"	"

Table 10 Page 7 of 36  
ANALYTICAL METHOD: WATER  
VOLATILES

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
toluene	602	1	624	10	WA 85-177	10	"	"	"
total xylenes	601	1	624	10	WA 85-177	10	"	"	"
trichloroethene	601	1	624	10	WA 85-177	10	"	"	"
trichlorofluoromethane	601	1	624	10	WA 85-177	10	"	"	"
vinyl acetate			624	10	WA 85-177	10	"	"	"
vinyl chloride	601	1	624	10	WA 85-177	10	"	"	"

Table 10 Page 8 of 36  
ANALYTICAL METHOD: WATER  
WET CHEMISTRY

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
	*	ppb		ppb		ppb	SEE BELOW	SEE BELOW	SEE BELOW
ammonia nitrogen	350.1	10					"	"	"
cyanide	335.2	50					"	"	"
nitrate + nitrite as N	353.1	10					"	"	"
nitrate nitrogen	350.1	10					"	"	"
percent solids	160.3	0.1					"	"	"
pH	150.1	0.1					"	"	"
specific conductance	120.1	0.1					"	"	"
total kjeldahl nitrogen	351.2	100					"	"	"
total organic carbon	415.1	1000					"	"	"
total organic halides	450.1	10					"	"	"
total phosphorus	365.4	10					"	"	"

\* - Methods Reference: EPA-600/4-79-020 "Methods for Chemical Analysis of Water and Waste Waters"

Table 10 Page 9 of 36  
ANALYTICAL METHOD: WATER  
METALS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
	#	ppb		ppb		ppb	SEE BELOW	SEE BELOW	SEE BELOW
aluminum	202.1	100	ICP	50			"	"	"
antimony	204.1	100	ICP	100			"	"	"
arsenic	206.2	1	ICP	100			"	"	"
barium	208.1	100	ICP	5			"	"	"
beryllium	210.1	10	ICP	5			"	"	"
cadmium	213.2	10	ICP	5			"	"	"
calcium	215.1	50	ICP	1000			"	"	"
chromium	218.1	10	ICP	10			"	"	"
cobalt	219.1	50	ICP	40			"	"	"
copper	220.1	10	ICP	10			"	"	"
iron	236.1	10	ICP	200			"	"	"
lead	239.1	10	ICP	100			"	"	"
magnesium	242.1	10	ICP	1000			"	"	"
manganese	243.1	10	ICP	10			"	"	"
mercury (cold vapor)	245.1	0.5	AA	.1			"	"	"
molybdenum	246.1	100	ICP	30			"	"	"
nickel	249.1	10	ICP	20			"	"	"
potassium	253.1	10	AA	1000			"	"	"
selenium	270.2	1	ICP	200			"	"	"
silver	272.1	10	ICP	10			"	"	"
sodium	273.1	10	ICP	1000			"	"	"
tin	282.1	500	ICP	100			"	"	"
titanium	283.1	1000	ICP	10			"	"	"
vanadium	285.1	1000	ICP	10			"	"	"
zinc	289.1	10	ICP	10			"	"	"

\* - Methods reference: A.A. by direct aspiration or A.A. furnace, EPA-600/4-79-220 "Method for Chemical Analysis of Water and Waste Water"

Table 10 Page 10 of 36  
ANALYTICAL METHOD: SOIL/SEDIMENT  
ACID EXTRACTABLES

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
	(GC)	ppb	(GC/MS)	ppb	CLP	ppb			
2,4,6-trichlorophenol	604	330	625	330	WA 85-177	330	SEE BELOW	SEE BELOW	SEE BELOW
2,4-dichlorophenol	604	330	625	330	WA 85-177	330	"	"	"
2,4-dimethylphenol	604	330	625	330	WA 85-177	330	"	"	"
2,4-dinitrophenol	604	1650	625	1650	WA 85-177	1600	"	"	"
2-chlorophenol	604	330	625	330	WA 85-177	330	"	"	"
2-fluorophenol	604	330	625	330	WA 85-177	330	"	"	"
2-methyl-4,6-dinitrophenol	604	660	625	660	WA 85-177	1600	"	"	"
2-nitrophenol	604	660	625	660	WA 85-177	330	"	"	"
4-chloro-3-methylphenol	604	330	625	330	WA 85-177	330	"	"	"
4-nitrophenol	604	1650	625	1650	WA 85-177	1600	"	"	"
Acid Extractable Screen							"	"	"
pentachlorophenol	604	1650	625	1650	WA 85-177	1600	"	"	"
pentafluorophenol	604	660	625	660	WA 85-177	1600	"	"	"
phenol	604	330	625	330	WA 85-177	330	"	"	"

Table 10 Page 11 of 36  
ANALYTICAL METHOD: SOIL/SEDIMENT  
BASE/NEUTRALS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
	(GC)	ppb	(GC/MS)	ppb	(CLP)	ppb			
							SEE BELOW	SEE BELOW	SEE BELOW
1,2,4-trichlorobenzene	612	330	625	330	WA 85-177	330	"	"	"
1,2-dichlorobenzene	602	330	625	330	WA 85-177	330	"	"	"
1,2-diphenylhydrazine	-	-	625	660	WA 85-177	330	"	"	"
1,3-dichlorobenzene	602	330	625	330	WA 85-177	330	"	"	"
1,4-dichlorobenzene	602	330	625	330	WA 85-177	330	"	"	"
2,4-dinitrotoluene	609	330	625	330	WA 85-177	330	"	"	"
2,6-dinitrotoluene	609	330	625	330	WA 85-177	330	"	"	"
2-chloronaphthalene	612	330	625	330	WA 85-177	330	"	"	"
3,3-dichlorobenzidine	605	1650	625	1650	WA 85-177	330	"	"	"
4-bromophenyl phenyl ether	611	330	625	330	WA 85-177	330	"	"	"
4-chlorophenyl phenyl ether	611	330	625	330	WA 85-177	330	"	"	"
acenaphthalene	610	330	625	330	WA 85-177	330	"	"	"
acenaphthene	610	330	625	330	WA 85-177	330	"	"	"
anthracene	610	330	625	330	WA 85-177	330	"	"	"
Base/Neutral Screen							"	"	"
benzidine	605	1650	625	1650	WA 85-177	330	"	"	"
benzo(a)anthracene	610	330	625	330	WA 85-177	330	"	"	"
benzo(a)pyrene	610	330	625	330	WA 85-177	330	"	"	"
benzo(b)fluoranthene	610	330	625	330	WA 85-177	330	"	"	"
benzo(g,h,i)perylene	610	660	625	660	WA 85-177	330	"	"	"
benzo(k)fluoranthene	610	330	625	330	WA 85-177	330	"	"	"
bis(2-chloroethoxy)methane	611	330	625	330	WA 85-177	330	"	"	"
bis(2-chloroethyl) ether	611	330	625	330	WA 85-177	330	"	"	"
bis(2-chloroisopropyl) ether	611	330	625	330	WA 85-177	330	"	"	"
bis(2-ethylhexyl) phthalate	606	330	625	330	WA 85-177	330	"	"	"
butyl benzyl phthalate	606	330	625	330	WA 85-177	330	"	"	"
chrysene	610	330	625	330	WA 85-177	330	"	"	"
di-n-butylphthalate	606	330	625	330	WA 85-177	330	"	"	"
di-n-octyl phthalate	606	330	625	330	WA 85-177	330	"	"	"
dibenzo(a,b)anthracene	610	660	625	660	WA 85-177	330	"	"	"



Table 10 Page 12 of 36  
ANALYTICAL METHOD: SOIL/SEDIMENT  
BASE/NEUTRALS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
diethyl phthalate	606	330	625	330	WA 85-177	330	"	"	"
dimethyl phthalate	606	330	625	330	WA 85-177	330	"	"	"
fluoranthene	610	330	625	330	WA 85-177	330	"	"	"
fluorene	610	330	625	330	WA 85-177	330	"	"	"
hexachlorobenzene	612	330	625	330	WA 85-177	330	"	"	"
hexachlorobutadiene	612	330	625	330	WA 85-177	330	"	"	"
hexachlorocyclopentadiene	612	330	625	330	WA 85-177	330	"	"	"
hexachlorocelhare	612	330	625	330	WA 85-177	330	"	"	"
indeno(1,2,3-c,d)pyrene	610	660	625	660	WA 85-177	330	"	"	"
isophorone	609	330	625	330	WA 85-177	330	"	"	"
N-nitrosodim-n-propylamine	607	660	625	660	WA 85-177	330	"	"	"
N-nitrosodiethylamine	607	1650	625	1650	WA 85-177	330	"	"	"
N-nitrosodiphenylamine	607	660	625	660	WA 85-177	330	"	"	"
naphthalene	610	330	625	330	WA 85-177	330	"	"	"
nitrobenzene	609	330	625	330	WA 85-177	330	"	"	"
phenanthrene	610	330	625	330	WA 85-177	330	"	"	"
pyrene	610	330	625	330	WA 85-177	330	"	"	"

Table 10 Page 13 of 36  
ANALYTICAL METHOD: SOIL/SEDIMENT  
DIOXINS/FURANS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
		ppb		ppt		ppb			
							SEE BELOW	SEE BELOW	SEE BELOW
tetra-CDD			SEE NOTE 5	20			"	"	"
tetra-CDF			SEE NOTE 5	20			"	"	"
penta-CDD			SEE NOTE 5	20			"	"	"
penta-CDF			SEE NOTE 5	20			"	"	"
hexa-CDD			SEE NOTE 5	20			"	"	"
hexa-CDF			SEE NOTE 5	20			"	"	"
hepta-CDD			SEE NOTE 5	200			"	"	"
hepta-CDF			SEE NOTE 5	200			"	"	"
octa-CDD			SEE NOTE 5	200			"	"	"
octa-CDF			SEE NOTE 5	200			"	"	"

NOTE 5 Reference Columbia National Fisheries Research lab procedure copy in original scope of services.

Table 10 Page 14 of 36  
ANALYTICAL METHOD: SOIL/SEDIMENT  
EXPLOSIVES

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
		ppb		ppb		ppb			
1,3 DNB			SEE NOTE 6	500					
1,3,5 TNB			SEE NOTE 6	500					
2,4 DNT			SEE NOTE 6	500					
2,4,6 TNT			SEE NOTE 6	500					
2,6 DNT			SEE NOTE 6	500					
HMX			SEE NOTE 6	500					
NB			SEE NOTE 6	500					
RDX			SEE NOTE 6	500					
tetryl			SEE NOTE 6	500					

NOTE 6 - USAMRIID Method 20: Cyclotrimethylenetrinitramine (RDX) in soil and sediment samples, 12/8/80.

Table 10 Page 15 of 36  
ANALYTICAL METHOD: SOIL/SEDIMENT  
METALS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
	μ	ppb		ppb		ppb			
aluminum	202.1	10000	ICP	5000			SEE BELOW	SEE BELOW	SEE BELOW
antimony	204.1	10000	ICP	10000			"	"	"
arsenic	205.2	100	ICP	10000			"	"	"
barium	208.1	10000	ICP	500			"	"	"
beryllium	210.1	1000	ICP	500			"	"	"
cadmium	213.2	1000	ICP	500			"	"	"
calcium	215.1	5000	ICP	100000			"	"	"
chromium	218.1	1000	ICP	1000			"	"	"
cobalt	219.1	5000	ICP	4000			"	"	"
copper	220.1	1000	ICP	1000			"	"	"
iron	235.1	1000	ICP	20000			"	"	"
lead	239.1	1000	ICP	10000			"	"	"
magnesium	242.1	1000	ICP	100000			"	"	"
manganese	243.1	1000	ICP	1000			"	"	"
mercury (cold vapor)	245.5	50	AA	10			"	"	"
molybdenum	246.1	10000	ICP	3000			"	"	"
nickel	249.1	1000	ICP	2000			"	"	"
potassium	258.1	1000	AA	100000			"	"	"
selenium	270.2	100	ICP	20000			"	"	"
silver	272.1	1000	ICP	1000			"	"	"
sodium	273.1	1000	ICP	100000			"	"	"
tin	282.1	50000	ICP	10000			"	"	"
titanium	283.1	100000	ICP	1000			"	"	"

Table 10 Page 16 of 36  
ANALYTICAL METHOD: SOIL/SEDIMENT  
METALS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
vanadium	285.1	100000	ICP	1000			"	"	"
zinc	289.1	1000	ICP	1000			"	"	"

\* -- Methods Reference: A.A. by direct aspiration or A.A. furnace, EPA-600/4-79-020 "Method for Chemical Analysis of Water and Waste"  
Reference Test Methods for Evaluating Solid Waste, EPA SW 846, Section 3050 (Revised 4/84).

Procedure - HCL final reflux for furnace, Sb, Sn

HCL final reflux for flame, Al, Sb, Ba, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Ni, K, Ag, Na, Ti, Sr, V, Zn

HNO<sub>3</sub> final reflux for furnace metals As, Be, Cd, Cr, Co, Cu, Fe, Pb, Mn, Ni, Se, Ag, Ti, V, Zn

Table 10 Page 17 of 36  
ANALYTICAL METHOD: SOIL/SEDIMENT  
PESTICIDES/PCBs

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
	(GC)	ppb	(GC/MS)	ppb	(CLP)	ppb			
							SEE BELOW	SEE BELOW	SEE BELOW
4,4'-DDD	608	4	625	330	WA 85-177	16	"	"	"
4,4'-DDE	608	4	625	330	WA 85-177	16	"	"	"
4,4'-DDT	608	4	625	330	WA 85-177	16	"	"	"
aldrin	608	2	625	330	WA 85-177	8	"	"	"
Aroclor 1015	608	17	625	330	WA 85-177	80	"	"	"
Aroclor 1221	608	17	625	330	WA 85-177	80	"	"	"
Aroclor 1232	608	17	625	330	WA 85-177	80	"	"	"
Aroclor 1242	608	17	625	330	WA 85-177	80	"	"	"
Aroclor 1248	608	17	625	330	WA 85-177	80	"	"	"
Aroclor 1254	608	33	625	330	WA 85-177	160	"	"	"
Aroclor 1260	608	33	625	330	WA 85-177	160	"	"	"
chlordane	608	17	625	330	WA 85-177	80	"	"	"
dieldrin	608	33	625	330	WA 85-177	16	"	"	"
endosulfan I	608	2	625	330	WA 85-177	8	"	"	"
endosulfan II	608	33	625	330	WA 85-177	16	"	"	"
endosulfan sulfate	608	33	625	330	WA 85-177	16	"	"	"
endrin	608	33	625	330	WA 85-177	16	"	"	"
endrin aldehyde	608	33	625	330	WA 85-177	16	"	"	"
endrin ketone	608	33	625	330	WA 85-177	16	"	"	"
heptachlor	608	2	625	330	WA 85-177	8	"	"	"
heptachlor epoxide	608	2	625	330	WA 85-177	8	"	"	"
methoxychlor	608	17	625	330	WA 85-177	80	"	"	"
toxaphene	608	33	625	330	WA 85-177	160	"	"	"
$\beta$ -BHC	608	2	625	330	WA 85-177	8	"	"	"
$\gamma$ -BHC (lindane)	608	2	625	330	WA 85-177	8	"	"	"
$\alpha$ -BHC	608	2	625	330	WA 85-177	8	"	"	"

Table 10 Page 18 of 36  
ANALYTICAL METHOD: SOIL/SEDIMENT  
VOLATILES

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
	(GC)	ppb	(GC/MS)	ppb	(CLP)	ppb			
							SEE BELOW	SEE BELOW	SEE BELOW
1,1,1-trichloroethane	601	1	624	10	WA 85-177	10	"	"	"
1,1,2,2-tetrachloroethane	601	1	624	10	WA 85-177	10	"	"	"
1,1,2-trichloroethane	601	1	624	10	WA 85-177	10	"	"	"
1,1-dichloroethane	601	1	624	10	WA 85-177	10	"	"	"
1,1-dichloroethene	601	1	624	10	WA 85-177	10	"	"	"
1,2-dichloropropane	601	1	624	10	WA 85-177	10	"	"	"
2-bromo-1-chloropropane	601	1	624	10	WA 85-177	10	"	"	"
2-butanone	602	10	624	100	WA 85-177	10	"	"	"
2-chloroethylethyl ether	601	1	624	10	WA 85-177	10	"	"	"
2-hexanone	602	10	624	100	WA 85-177	10	"	"	"
4-methyl-2-pentanone		10	624	100	WA 85-177	10	"	"	"
acetone	601	10	624	100	WA 85-177	10	"	"	"
benzene	602	1	624	10	WA 85-177	10	"	"	"
bromochloromethane	601	1	624	10	WA 85-177	10	"	"	"
bromodichloromethane	601	1	624	10	WA 85-177	10	"	"	"
bromoform	601	10	624	10	WA 85-177	10	"	"	"
bromomethane	601	10	624	100	WA 85-177	10	"	"	"
c-1,3-dichloropropene	601	1	624	10	WA 85-177	10	"	"	"
carbon tetrachloride	601	1	624	10	WA 85-177	10	"	"	"
chlorobenzene	602	1	624	10	WA 85-177	10	"	"	"
chloroethane	601	10	624	100	WA 85-177	10	"	"	"
chloroform	601	1	624	10	WA 85-177	10	"	"	"
chloromethane	601	10	624	100	WA 85-177	10	"	"	"
dibromochloromethane	601	1	624	10	WA 85-177	10	"	"	"
dichlorodifluoromethane	601	1	624	10	WA 85-177	10	"	"	"
ethyl benzene	602	1	624	10	WA 85-177	10	"	"	"
methylene chloride	601	1	624	10	WA 85-177	10	"	"	"
t-1,1-trichloroethene	601	1	624	10	WA 85-177	10	"	"	"
t-1,3-dichloropropene	601	1	624	10	WA 85-177	10	"	"	"
tetrachloroethene	601	1	624	10	WA 85-177	10	"	"	"

Table 10 Page 19 of 36  
ANALYTICAL METHOD: SOIL/SEDIMENT  
VOLATILES

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
toluene	602	1	624	10	WA 85-177	10	"	"	"
total xylenes	601	1	624	10	WA 85-177	10	"	"	"
trichloroethene	601	1	624	10	WA 85-177	10	"	"	"
trichlorofluoromethane	601	1	624	10	WA 85-177	10	"	"	"
vinyl			624	10	WA 85-177	10	"	"	"
vinyl chloride	601	1	624	10	WA 85-177	10	"	"	"



Table 10 Page 20 of 36  
ANALYTICAL METHOD: SOIL/SEDIMENT  
WET CHEMISTRY

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
	*	ppb		ppb					
							SEE BELOW	SEE BELOW	SEE BELOW
ammonia nitrogen	350.1	1000	SEE NOTE 1				"	"	"
cation exchange capacity	9080						"	"	"
cyanide	335.2	5000	SEE NOTE 1				"	"	"
nitrate + nitrite as N	353.1	1000	" " "				"	"	"
nitrate nitrogen	350.1	1000	" " "				"	"	"
percent solids	160.3	0.1					"	"	"
pH	150.1	10	SEE NOTE 1				"	"	"
specific conductance	120.1	10	SEE NOTE 1				"	"	"
total Kjeldahl nitrogen	351.2	10000	SEE NOTE 2				"	"	"
total organic carbon	415.1	100000	SEE NOTE 1				"	"	"
total organic halides	450.1	1000	SEE NOTE 3				"	"	"
total phosphorus	365.4	1000	SEE NOTE 2				"	"	"

\* - Method Reference: EPA-600/4-73-020 "Methods for Chemical Analysis of Water and Waste"

NOTE 1 SLUDGE/SOIL/SEDIMENT Aliquot are extracted with distilled deionized water for 24 hours and the supernant is analyzed by the referenced aqueous procedure

NOTE 2 A portion of the SLUDGE/SOIL/SEDIMENT is subjected to the block digester procedure referenced in aqueous procedure.

NOTE 3 A SLUDGE/SOIL/SEDIMENT sample is extracted with ethyl acetate and the extract is pyrolyzed for TOX.

Table 10 Page 21 of 36  
ANALYTICAL METHOD: BIOTA  
ACID EXTRACTABLES

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
2,4,6-trichlorophenol					SEE NOTE 12		"	"	"
2,4-dichlorophenol					SEE NOTE 12		"	"	"
2,4-dimethylphenol					SEE NOTE 12		"	"	"
2,4-dinitrophenol					SEE NOTE 12		"	"	"
2-chlorophenol					SEE NOTE 12		"	"	"
2-fluorophenol					SEE NOTE 12		"	"	"
2-methyl-4,6-dinitrophenol					SEE NOTE 12		"	"	"
2-nitrophenol					SEE NOTE 12		"	"	"
4-chloro-3-methylphenol					SEE NOTE 12		"	"	"
4-nitrophenol					SEE NOTE 12		"	"	"
Acid Extractable Screen					SEE NOTE 12		"	"	"
pentachlorophenol					SEE NOTE 12		"	"	"
pentafluorophenol					SEE NOTE 12		"	"	"
phenol					SEE NOTE 12		"	"	"

NOTE 12      Sampling and Analysis Procedures for Surveying of Fish for Priority Pollutants USEPA June 1977  
Metal Detection Limits are the same as for Soil/Sediment  
Detection Limits Organics - 50 µg./Kg.  
Cd, Pb, - Flame  
Hg - Cold Vapor

Table 10 Page 22 of 36  
ANALYTICAL METHOD: BIOTA  
BASE/NEUTRALS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
							SEE BELOW	SEE BELOW	SEE BELOW
1,2,4-trichlorobenzene					SEE NOTE 12		"	"	"
1,2-dichlorobenzene					SEE NOTE 12		"	"	"
1,2-diphenylhydrazine					SEE NOTE 12		"	"	"
1,3-dichlorobenzene					SEE NOTE 12		"	"	"
1,4-dichlorobenzene					SEE NOTE 12		"	"	"
2,4-dinitrotoluene					SEE NOTE 12		"	"	"
2,6-dinitrotoluene					SEE NOTE 12		"	"	"
2-chloronaphthalene					SEE NOTE 12		"	"	"
3,3-dichlorobenzidine					SEE NOTE 12		"	"	"
4-bromophenyl phenyl ether					SEE NOTE 12		"	"	"
4-chlorophenyl phenyl ether					SEE NOTE 12		"	"	"
acenaphthalene					SEE NOTE 12		"	"	"
acenaphthene					SEE NOTE 12		"	"	"
anthracene					SEE NOTE 12		"	"	"
Base/Neutral Screen					SEE NOTE 12		"	"	"
benzidine					SEE NOTE 12		"	"	"
benzo(a)anthracene					SEE NOTE 12		"	"	"
benzo(a)pyrene					SEE NOTE 12		"	"	"
benzo(b)fluoranthene					SEE NOTE 12		"	"	"
benzo(h,h <sub>1</sub> )perylene					SEE NOTE 12		"	"	"
benzo(k)fluoranthene					SEE NOTE 12		"	"	"
bis(2-chloroethoxy) methane					SEE NOTE 12		"	"	"
bis(2-chloroethyl) ether					SEE NOTE 12		"	"	"
bis(2-chloroisopropyl) ether					SEE NOTE 12		"	"	"
bis(2-ethylhexyl)phthalate					SEE NOTE 12		"	"	"
butyl benzyl phthalate					SEE NOTE 12		"	"	"
chrysene					SEE NOTE 12		"	"	"
di-n-butylphthalate					SEE NOTE 12		"	"	"
di-n-octyl phthalate					SEE NOTE 12		"	"	"
dibenzo(a,n)anthracene					SEE NOTE 12		"	"	"
diethyl phthalate					SEE NOTE 12		"	"	"

Table 10 Page 23 of 36  
ANALYTICAL METHOD: BIOTA  
BASE/NEUTRALS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
dimethyl phthalate					SEE NOTE 12		"	"	"
fluoranthene					SEE NOTE 12		"	"	"
fluorene					SEE NOTE 12		"	"	"
hexachlorobenzene					SEE NOTE 12		"	"	"
hexachlorobutadiene					SEE NOTE 12		"	"	"
hexachlorocyclopentadiene					SEE NOTE 12		"	"	"
hexachloroethane					SEE NOTE 12		"	"	"
indeno (1,2,3-c,d) pyrene					SEE NOTE 12		"	"	"
isophorone					SEE NOTE 12		"	"	"
N-nitrosodimethylamine					SEE NOTE 12		"	"	"
N-nitrosodimethylamine					SEE NOTE 12		"	"	"
N-nitrosodiphenylamine					SEE NOTE 12		"	"	"
naphthalene					SEE NOTE 12		"	"	"
nitrobenzene					SEE NOTE 12		"	"	"
phenanthrene					SEE NOTE 12		"	"	"
pyrene					SEE NOTE 12		"	"	"

NOTE 12      Sampling and Analysis Procedures for Surveying of Fish for Priority Pollutants USEPA June 1977  
Metal Detection Limits are the same as for Soil/Sediment  
Detection Limits Organics - 50 µg./Kg.  
Cd, Pb, - Flame  
Hg - Cold Vapor

Table JO Page 24 of 36  
ANALYTICAL METHOD: BIOTA  
DIOXINS/FURANS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
tetra-CDD					SEE NOTE 12		SEE BELOW	SEE BELOW	SEE BELOW
tetra-CDF					SEE NOTE 12		"	"	"
penta-CDD					SEE NOTE 12		"	"	"
penta-CDF					SEE NOTE 12		"	"	"
hexa-CDD					SEE NOTE 12		"	"	"
hexa-CDF					SEE NOTE 12		"	"	"
hepta-CDD					SEE NOTE 12		"	"	"
hepta-CDF					SEE NOTE 12		"	"	"
octa-CDD					SEE NOTE 12		"	"	"
octa-CDF					SEE NOTE 12		"	"	"

NOTE 12      Sampling and Analysis Procedures for Surveying of Fish for Priority Pollutants USEPA June 1977  
Metal Detection Limits are the same as for Soil/Sediment  
Detection Limits Organics - 50 µg./Kg.  
Cd, Pb, - Flame  
Hg - Cold Vapor

Table 10 Page 25 of 36  
ANALYTICAL METHOD: BIOTA  
METALS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
cadmium					SEE NOTE 12		SEE BELOW	SEE BELOW	SEE BELOW
lead					SEE NOTE 12		"	"	"
mercury (cold vapor)					SEE NOTE 12		"	"	"

NOTE 12      Sampling and Analysis Procedures for Surveying of Fish for Priority Pollutants USEPA June 1977  
Metal Detection Limits are the same as for Soil/Sediment  
Detection Limits Organics - 50 µg./Kg.  
Cd, Pb, - Flame  
Hg - Cold Vapor

Table 10 Page 26 of 36  
ANALYTICAL METHOD: BIOTA  
PEST ICIDES/PCRS

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
							SEE BELOW	SEE BELOW	SEE BELOW
4,4'-DDD					SEE NOTE 12		"	"	"
4,4'-DDE					SEE NOTE 12		"	"	"
4,4'-DDT					SEE NOTE 12		"	"	"
aldrin					SEE NOTE 12		"	"	"
Aroclor 1248					SEE NOTE 12		"	"	"
Aroclor 1231					SEE NOTE 12		"	"	"
Aroclor 1232					SEE NOTE 12		"	"	"
Aroclor 1242					SEE NOTE 12		"	"	"
Aroclor 1248					SEE NOTE 12		"	"	"
Aroclor 1254					SEE NOTE 12		"	"	"
Aroclor 1260					SEE NOTE 12		"	"	"
chlordane					SEE NOTE 12		"	"	"
dieldrin					SEE NOTE 12		"	"	"
endosulfan I					SEE NOTE 12		"	"	"
endosulfan II					SEE NOTE 12		"	"	"
endosulfan sulfate					SEE NOTE 12		"	"	"
endrin					SEE NOTE 12		"	"	"
endrin aldehyde					SEE NOTE 12		"	"	"
endrin ketone					SEE NOTE 12		"	"	"
heptachlor					SEE NOTE 12		"	"	"
heptachlor epoxide					SEE NOTE 12		"	"	"
methoxychlor					SEE NOTE 12		"	"	"
toxaphene					SEE NOTE 12		"	"	"
$\beta$ -BHC					SEE NOTE 12		"	"	"

Table 10 Page 27 of 36  
ANALYTICAL METHOD: BIOTA  
PESTICIDES/PCBs

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
$\gamma$ -BHC (lindane)					SEE NOTE 12		"	"	"
$\alpha$ -BHC					SEE NOTE 12		"	"	"

NOTE 12      Sampling and Analysis Procedures for Surveying of Fish for Priority Pollutants USEPA June 1977  
Metal Detection Limits are the same as for Soil/Sediment  
Detection Limits Organics - 50  $\mu\text{g.}/\text{Kg.}$   
Cd, Pb, - Flame  
Hg - Cold Vapor



Table 10 Page 28 of 36  
ANALYTICAL METHOD: BIOTA  
VOLATILES

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
							SEE BELOW	SEE BELOW	SEE BELOW
1,1,1-trichloroethane					SEE NOTE 12		"	"	"
1,1,2,2-tetrachloroethane					SEE NOTE 12		"	"	"
1,1,2-trichloroethane					SEE NOTE 12		"	"	"
1,1-dichloroethane					SEE NOTE 12		"	"	"
1,1-dichloroethene					SEE NOTE 12		"	"	"
1,2-dichloropropane					SEE NOTE 12		"	"	"
2-bromo-1-chloropropane					SEE NOTE 12		"	"	"
2-butanone					SEE NOTE 12		"	"	"
2-chloroethylvinyl ether					SEE NOTE 12		"	"	"
2-hexanone					SEE NOTE 12		"	"	"
4-methyl-2-pentanone					SEE NOTE 12		"	"	"
acetone					SEE NOTE 12		"	"	"
benzene					SEE NOTE 12		"	"	"
bromochloromethane					SEE NOTE 12		"	"	"
bromodichloromethane					SEE NOTE 12		"	"	"
bromoform					SEE NOTE 12		"	"	"
bromomethane					SEE NOTE 12		"	"	"
c-1,3-dichloropropene					SEE NOTE 12		"	"	"
carbon tetrachloride					SEE NOTE 12		"	"	"
chlorobenzene					SEE NOTE 12		"	"	"
chloroethane					SEE NOTE 12		"	"	"
chloroform					SEE NOTE 12		"	"	"
chloromethane					SEE NOTE 12		"	"	"
dibromochloromethane					SEE NOTE 12		"	"	"
dichlorodifluoromethane					SEE NOTE 12		"	"	"
ethyl benzene					SEE NOTE 12		"	"	"
methylene chloride					SEE NOTE 12		"	"	"
t-1,1-trichloroethene					SEE NOTE 12		"	"	"
t-1,3-dichloropropene					SEE NOTE 12		"	"	"
tetrachloroethene					SEE NOTE 12		"	"	"
toluene					SEE NOTE 12		"	"	"

Table 10 Page 29 of 36  
ANALYTICAL METHOD: BIOTA  
VOLATILES

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
total xylenes					SEE NOTE 12		"	"	"
trichloroethene					SEE NOTE 12		"	"	"
trichlorofluoromethane					SEE NOTE 12		"	"	"
vinyl acetate					SEE NOTE 12		"	"	"
vinyl chloride					SEE NOTE 12		"	"	"

NOTE 12      Sampling and Analysis Procedures for Surveying of Fish for Priority Pollutants USEPA June 1977  
Metal Detection Limits are the same as for Soil/Sediment  
Detection Limits Organics - 50 µg./Kg.  
Cd, Pb, - Flame  
Hg - Cold Vapor

Table 10 Page 30 of 36  
ANALYTICAL METHOD: BIOTA  
WET CHEMISTRY

CHEMICAL COMPOUND	METHOD 1	DETECTION LIMIT 1	METHOD 2	DETECTION LIMIT 2	METHOD 3	DETECTION LIMIT 3	AUDIT	FREQUENCY	CONTROL LIMITS
							SEE BELOW	SEE BELOW	SEE BELOW
ammonia nitrogen					SEE NOTE 12		"	"	"
cyanide					SEE NOTE 12		"	"	"
nitrate + nitrite as N					SEE NOTE 12		"	"	"
nitrate nitrogen					SEE NOTE 12		"	"	"
percent solids					SEE NOTE 12		"	"	"
pH					SEE NOTE 12		"	"	"
specific conductance					SEE NOTE 12		"	"	"
total Kjeldahl nitrogen					SEE NOTE 12		"	"	"
total organic carbon					SEE NOTE 12		"	"	"
total organic halides					SEE NOTE 12		"	"	"
total phosphorus					SEE NOTE 12		"	"	"

NOTE 12      Sampling and Analysis Procedures for Surveying of Fish for Priority Pollutants USEPA June 1977  
Metal Detection Limits are the same as for Soil/Sediment  
Detection Limits Organics - 50 µg./Kg.  
Cd, Pb, - Flame  
Hg - Cold Vapor

PESTICIDES/PCBs  
CRAB ORCHARD NATIONAL WILDLIFE REFUGE

AUDIT	FREQUENCY	CONTROL LIMITS
Retention Time Windows	Once per 24 hours	4,4'-DDT must have retention time greater than or equal to 12 minutes on packed column, less than 2% shift on packed and .3% for capillary column.
Evaluation Mixtures A, B, & C	Once per 72 hours.	% RSD for aldrin, endrin & dibutylchloroendate must be less than or equal to 10%.
Column Breakthrough	Once per 72 hours.	Must not exceed 20% - if greater remedial action is required.
Industrial Standard Mix	Once per 72 hours then intermittently throughout analysis	Calculated factors must not exceed 15% difference for the quantitation run nor 20% difference for confirmation run during 12 hour period. Deviation greater than or equal to 15% requires reanalysis.
Confirmation Analysis	Once per 72 hours.	Separation should be greater than or equal to 25% resolution between peaks.
Reagent Blank	1 per case or 1 in 20 of similar concentration/matrix	Less than 5x CRDL for solvents, less than CRDL for all others.
Surrogate Spike	All samples and blank (including MS/MSD).	Recovery limits within those of Table 4.2, Exhibit E (IFB WA 85-J176, WA 85-J177, WA 85-178 (revised 1/85)).
MS/MSD	1 per case or 1 in 20 of similar concentration/matrix	Must fall within limits of Table 5.2, Exhibit E (IFB WA 85-J176, WA 85-J177, WA 85-178 (revised 1/85)).

VOLATILES  
CRAB ORCHARD NATIONAL WILDLIFE REFUGE

AUDIT	FREQUENCY	CONTROL LIMITS
Reagent Blank	1 per case or 1 in 20 of similar concentration/matrix.	Less than 5x CRDL for solvents, less than CRDL for all others.
Surrogate Spike	All samples and blank (including ms/msd).	Recovery limits within those of Table 4.2, Exhibit E (JFB WA 85-J176, WA 85-J177, WA 85-178 (revised 1/85)).
MS/MSD	1 per case or 1 in 20 of similar concentration/matrix.	Recovery limits within those of Table 5.2, Exhibit E (JFB WA 85-J176, WA 85-J177, WA 85-178 (revised 1/85)).
Calibration Continuing	Each 12 hours	Minimum RF 0.003; must be less than 25% difference for any check compound.
Method/Field Blank	1 in 20-provided by sampling crew	Same as reagent blank
Replicate	1 in 20-provided by sampling crew	+/- 20% PRE waters +/- 50% PRE soils
MS Tuning	One per day.	BFB key ions and abundance criteria must be met for all 9 ions.
Calibration Verification	Once	Five concentrations - linear range Base/Neutrals 0-400 ng. Acids 0-1000 ng.

SEMI-VOLATILES  
CRAB ORCHARD NATIONAL WILDLIFE REFUGE

AUDIT	FREQUENCY	CONTROL LIMITS
Reagent Blank	1 per case or 1 in 20 of similar concentration/matrix.	Less than 5x CRDL for solvents, less than CRDL for all others.
Surrogate Spike	All samples and blank (including MS/MSD).	Recovery limits within those of Table 4.2, Exhibit E (IFB WA 85-J176, WA 85-J177, WA 85-178 (revised 1/85)).
MS/MSD	1 per case or 1 in 20 of similar concentration/matrix.	Recovery limits within those of Table 5.2, Exhibit E (IFB WA 85-J176, WA 85-J177, WA 85-178 (revised 1/85)).
Calibration Continuing	Each 12 hours	Minimum RF 0.05; must be less than 25% difference for any check compound.
Method/Field Blank	1 in 20-provided by sampling crew	Same as reagent blank
Replicate	1 in 20-provided by sampling crew	+/- 20% PRE waters +/- 50% PRE soils
MS Tuning	One per day.	BFB key ions and abundance criteria must be met for all 9 ions.
Calibration Verification	Once	Five concentrations - linear range Base/Neutrals 0-400 ng. Acids 0-1000 ng.

INORGANICS  
CRAB ORCHARD NATIONAL WILDLIFE REFUGE

AUDIT	FREQUENCY	CONTROL LIMITS
Calibration Verification	Calibrated daily and each time instrument is set up; verify at a frequency of 10% or every 2 hours, whichever is greater.	Within +/- 10% of true value for all except tin and mercury (+/- 20% of true value)
Calibration Blank	During calibration at a frequency of 10% during run and at end of run.	No more than CRDL.
Preparation Blank	1 per batch of samples digested or 1 in 20 whichever is greater	No more than CRDL.
Interference Check Sample	at beginning and end of or twice per 8 hour working shift whichever is greater.	+/- 20% of mean value (established by running samples at least 5 times repetitively).
Spiked Sample Analysis	1 per group of similar concentration and matrix, 1 per case of samples, or 1 in 20, whichever is greater.	Within +/- 25% recovery
Duplicate Sample Analysis	Same as spiked sample analysis.	+/- 20% RPD for values 5X CRDL or more +/- CRDL for samples less than 5X CRDL
Lab Control Sample (aqueous)	1 for each procedure for each case of samples received; 1 in 20 or 1 per batch digested, whichever is greater.	Within 80-120% recovery
Lab Control Sample (soils)	once a month for each of the procedures (applied) to solid sample analysis.	Within limits established by EPA.
Duplicate Injection	each analysis	+/- 20% RSD
Spike Sample	each analysis	In accordance with limits shown in Section 7, Exhibit E, SOW no. 784 (July 1984)

WET CHEMISTRY  
CRAB ORCHARD NATIONAL WILDLIFE REFUGE

AUDIT	FREQUENCY	CONTROL LIMITS
Calibration Verification	calibrated daily and each time instrument is set up; verify at a frequency of 10% or every 2 hours, whichever is greater.	Within +/- 10% of true value.
Calibration Blank	during calibration, at a frequency of 10% during run, and at end of run.	No more than CRDL
Preparation Blank	1 per batch of samples or 1 in 20, whichever is greater	No more than CRDL
Interference Check Sample	At beginning and end of each run or twice per 8 hour working shift.	+/- 20% of mean value (established by running sample at least 5 times repetitively); check sample to be prepared in consultation with EPA.
Duplicate Sample Analysis	1 per case of samples or 1 in 20 whichever is greater	+/- 20% RPD for values 5X CRDL or more; +/- CRDL for samples less than 5X CRDL
Spiked Sample Analysis	1 per group of similar concentration, 1 per case of samples, or 1 in 20; 1 at end of run for nitrate and nitrite.	within +/-25% recovery



ABBREVIATIONS USED IN TABLE 3

CRDL	-	CONTRACT REQUIRED DETECTION LIMITS
RF	-	RESPONSE FACTOR
PRE	-	PERCENT RELATIVE ERROR
MS/MSD	-	MATRIX SPIKE/MATRIX SPIKE DUPLICATE
RPD	-	RELATIVE PERCENT DIFFERENCE
RSD	-	RELATIVE STANDARD DEVIATION
TOX	-	TOTAL ORGANIC HALOGENS
ppb	-	PARTS PER BILLION
ppt	-	PARTS PER TRILLION
AA	-	ATOMIC ABSORPTION
C	-	GAS CHROMATOGRAPH
GC	-	GAS CHROMATOGRAPH/MASS SPECTROMETER
CLP	-	CONTRACT LABORATORY PROGRAM

reference standard samples, and/or field or laboratory blanks along with each set of samples. Any interference are identified and documented.

In general, the methods accuracy is determining by spiking the sample matrix with the analyte at a minimum of three concentration levels. The range of the spiking levels is selected to bracket the concentration of interest. Percent recoveries of the spikes are calculated and are compared with synthetic standards. The methods precision is determined by analyzing a minimum of three replicates at each spiking level. The precision is evaluated by calculating the standard deviation.

The data generated is, whenever possible, input the laboratory base data management system. Analyst's work sheets are filed for one year as a temporary record. When approved and signed, data reports and pertinent information are reported to the client.

The analytical protocols for explosives in soils are presented in Attachment 5. Samples to be analyzed for chlorinated dioxins and dibenzofurans will be analyzed according to the procedure of Smith et al. (1984) or equivalent as presented in Attachment 6.

## 7.02 Field Procedures

Site investigations will be conducted in two phases. Samples collected during the two phases will be shipped, following chain-of-custody procedures to O'Brien & Gere's laboratory for analyses.

Field analyses of surface and groundwater will consist of pH, specific conductance and temperature measurements.

*methods & calibration*

### VOLATILE ORGANICS IN WATER SAMPLES

Water samples were analyzed by EPA Methods 601 and 602 which employs the sample preparation step of purge and trap followed by analyses with a gas chromatograph equipped with a HECD and PID detector. Surrogate standards were added to a level of 20 ppb to each samples to monitor purging efficiency. The surrogate standards are:

Bromochloromethane

1,4 - Dichlorobutane

1 - Chloro - 2 - Brodopropane

### REFERENCE

- 1) Federal Register, 40 CFR, Part 136, October 26, 1984, Method 601 and 602.

## VOLATILE ORGANIC SCREENS IN SOIL AND SEDIMENT

Soil and sediment matrices were analyzed by a method which combined the sample preparation methods of the EPA CLP with the gas chromatographic methods of EPA Methods 601 and 602. The method employed a direct sparge sample preparation step followed by analysis with a gas chromatograph equipped with a HECD and a PID detector in series. Surrogate standards were added to every sample to monitor sparging efficiency. The surrogate standards are:

Bromochloromethane

1,4 - dichlorobutane

1 - chloro - 2 - Bromopropane

1000 ng of each surrogate was added to each soil/sediment sample.

## REFERENCE

- 1) Federal Register, 40 CFR, Part 136, October 26, 1984
- 2) USEPA Contract Laboratory Program, Statement of Work for Organic Analysis May, 1984

### SEMI VOLATILE FIDSCANS OF WATER AND SOIL SAMPLES

Water and soil samples were screened from organic pollutants using the sample preparation procedures of the EPA CLP. Identification and quantitation was accomplished using a gas chromatograph equipped with capillary column and a FID detector. Water samples were acid/base partitioned and extracted with methylene chloride. According to the CLP procedure, generating two extracts for analysis. Soil samples were extracted at a neutral pH as specified in the CLP, generating a single extract for analysis. Surrogate standards were added to all samples as follows:

	<u>Water</u>	<u>Soil</u>
Octadecane	200 ppb	6,700 ppb
2,4,6-Tribromo-phenol	500 ppb	17,000 ppb

The gas chromatograph conditions were as follows:

GC: HP 5880

Column: HP 530u Methyl silicone

Wide Bore Capillary

Column, 10 meters

Oven Temp: 50 to 250 at 10° per minute

### REFERENCES

- 1) USEPA Contract Laboratory Program Statement of Work for Organic Analysis 5/84

## ORGANO CHLORINE PESTICIDES AND PCBs IN WATER AND SOIL SAMPLES

The EPA CLP was used to screen samples for pesticides and PCBs in water and soil samples. Surrogate standards were added to every sample to monitor extraction efficiency. Dibutylchlorodate was the surrogate standard added to water samples at a level of 0.5 ppb and soil samples at 17 ppb.

### REFERENCES

- 1) USEPA Contract Laboratory Program Statement of Work for Organic Analysis May, 1984

## SECTION 8 - DATA REDUCTION, VALIDATION, AND REPORTING

### 8.01 General

O'Brien & Gere's laboratory facilities will perform all testing except for samples split with the U.S.F.W.S., explosives residues by HPLC, ICP scans, and PCDD/PCDF analyses. Data reduction and validation will be incorporated into the in-house effort.

### 8.02 Data Reduction and Reporting

The following data handling procedures are employed at O'Brien & Gere:

- A. Data Production - A Hewlett-Packard Model 5995 and 5993 are used for the positive identification and quantification of sample extracts. Output from the determination is a total ion chromatogram recorded on thermal printer hard copy and cassette tape.
- B. Data Reduction - Output from the GC/MS unit is digitized, stored in memory on cassette tape and processed for presentation in three formats:
  - 1) A real-time total multiple ion mass chromatogram.
  - 2) A post-run integration report contains the following:
    - a. Retention time
    - b. Response factor
    - c. Primary, secondary, and tertiary ion with their corresponding abundance
    - d. Quantitation ion

- e. Reference library name
  - f. Concentration
- 3) A visual comparison of the subject mass spectral output to the library compound.

C. Data Transcription - The post integration report contains the following:

- 1) Listing of all compounds.
- 2) Relative retention times.
- 3) Relative response factor to their internal standards.
- 4) Concentration of compounds, surrogate and internals.

Quality Control/Quality Assurance data such as resolution and calibration standards and DFTPP spectra are also processed and stored in the above manner.

D. Data Verification - The processed transcribed information and the hard copied raw data are now evaluated by the Group Leader to verify the validity of the data and determine whether reinjection or additional cleanup steps are required. The results of the evaluation are recorded in a notebook and inputted into the Sample Status File.

E. Distribution - Following final review the GC/MS Group Leader and Manager of Analytical Services, the results of the analytical determination are shipped to the Contractor. The format used for presentation of data are the presented in the IFB forms. Additional data such as copies of raw data and chromatograms are provided upon request.



F. In-House Storage - Results of all analytical determinations are stored in the RTE6 computer. Raw data tapes are logged into the computer on a separate file and listed by tape number and its contents. The data tapes are stored indefinitely. Should a request be made for a particular raw data tape, the tape is copied and the copy is kept in the archive while the original is sent to the Contractor. All notebooks are also archived and stored in the O'Brien & Gere Central File.

### Reporting

Once a sample has been tagged and input into the laboratory data management system, we have the ability to determine its exact status. With the available maintenance programs, and tracking forms, the group leaders can trace the progress of one sample or an entire group of samples. Therefore, a client is able to receive partial data before the entire program is complete.

For a program that covers the course of several months or years, it is imperative that interim reports be submitted. It is anticipated that turnaround for a batch of samples will be 40 days from sample arrival. The RTE6 computer system, with the Aquarius software will generate a final report following injection and data evaluation. Therefore, if specific sample information is required prior to submission of the case, we would be able to satisfy EPA's needs.

Of course there may be certain instances where faster turnaround would be dictated and we shall make every attempt to

meet those needs. Our past experience on programs of this size have proven our capability to supply information in a timely manner.

### 8.03 Data Validation

Prior to submitting of the data to the Project Manager for his review, data will be validated by the individual laboratory group leaders and/or Manager.

The validation process will include the review of spike recoveries, surrogate recovery, comparability of duplicate analysis and field blank integrity. Additionally, the reviewer will check for the adherence to accuracy and precision criteria, unusually high or low parameter values and possible transmittal errors.

Field data will be reviewed by the Quality Assurance Manager (QAM). The QAM will critique the field data using the same guidelines where required, as outlined above.

*Should we insist on use of  
Region V data validation  
procedures?  
or why really doing this?*

## SECTION 9 - INTERNAL QUALITY CONTROL PROCEDURES

### 9.01 Contract Laboratory Quality Control

The standard quality control procedures for the CLP will be employed to provide consistent, accurate and dependable test results.

Attachment 4 to this QAPP documents the QA/QC considerations included in this program. The major elements of the QA/QC program are: instrumental tuning and calibration criteria, defined analytical protocols, reagent blanks, surrogate spikes, matrix spikes and duplicate analyses. A reagent blank is included in each batch of up to twenty samples analyzed. Surrogate spike standards are incorporated into all samples and blanks prior to sample processing while one set of matrix spikes and matrix spike duplicates will be included per batch of up to twenty samples. A field blank consisting of diatomaceous earth for soils or distilled water for groundwater will also be included as quality control samples.

Sample containers will be supplied by the O'Brien & Gere's laboratory. In order to insure both sufficient quantity and proper container cleanliness the contract laboratory will order these supplies from I Chem Research, Inc. located in Hayward California. When ordering the containers the contract laboratory will specify pre-cleaned jars with teflon liners.

The types of containers are as follows:

#### A - Water

- °125 ml screwcap glass for teflon level
- °100 ml polyethylene bottle with screw cap
- °40 ml VOA vials with teflon septum

*clearing procedures.*

°500 ml polyethylene bottles with screw caps

°quart glass jars with teflon lined cap

°gallon glass jars with teflon lined cap

#### B - Soil/Sediment

°40 ml VOA vials with teflon system

°30 ml crimp vials *P*

°60 ml glass jars with teflon lined cap

°Wide mouth pint glass for with teflon lined cap

°1/2 pint glass for with teflon lined cap

In order to insure container cleanliness randomly selected containers will be filled with distilled deionized water and sent to the laboratory for analyses. The analyses requested for this blank sample will be equivalent to that for which the sample to be held by that container would normally be analyzed for.

#### 9.02 Field Sampling Quality Control

Field sampling crews will always be under the direct supervision of a crew chief with a minimum of a Bachelor's degree and five years sampling experience. New employees will be assigned to an experienced staff member and work under his/her direction.

Bound log books and appropriate data sheets will be used to document the collection of samples so that any individual sample can be traced back to its point of origin; sampler and sampling equipment.

Duplicate samples will be collected at the same time, employing the same procedures, equipment and containers as the scheduled sample.

Additionally, duplicate samples will be packaged and shipped to the laboratory in the same manner as the required sample.

Tables 6 and 7 (Section 1.05) list the projected number and sample type of duplicates for Phases I and II.

As specified in Section 8 of this QAPP the QAM will periodically review the results of the duplicate analyses and advise the Project Manager of any problems.

### 9.03 Field Analytical Procedures Quality Control

Field measurements of pH, temperature and specific conductance will be taken on water samples only. The pH meter will be checked against two known standard pH buffers (7 and 10) before and after each days use.

Temperature measurements will be made with a mercury-filled celsius thermometer. As a minimum, the thermometer will have a scale marked for every 0.1C, with marking etched on the capillary glass. Field operations will require a thermometer with a protective case to prevent breakage. The thermometer will be checked against a precision thermometer certified by the National Bureau of Standards (NBS) periodically.

Conductivity reading will be made with a portable specific conductivity meter. The meter will be calibrated against a 0.010 normal potassium chloride solution (KCL) at least once per day.

## SECTION 10 - AUDIT PROCEDURES

*Q10.2.1*  
The O'Brien & Gere Project Manager, the Columbia National Fisheries QC/QA Representative and the Refuge Manager will monitor and audit the performance of the QA procedures listed in this plan. They will conduct field and office audits.

*Do you have these results?*  
O'Brien & Gere has designated a QA office as indicated in Figure 3 (Section 2.02). A performance audit, consisting of analysis of appropriate blanks, fortified samples and standard solutions will be performed quarterly for the duration of the project. O'Brien & Gere's QA Officer will maintain a record of such audits and will inform the FWS of significant deviations from established control limits. These audits will test not only the total system's response, but inherently all major measurement methods.

*Is this being done?*  
O'Brien & Gere's QA Officer will report to the Project Manager and the FWS the result of assessment of: the accuracy, precision and completeness of the data, results of the performance and system audits, and any problems encountered in the analytical procedures. The QA Officer, in conjunction with the analyst, analyst's supervisor, and Project Manager will formulate recommendations to correct any deficiency in the analytical protocol or data. These corrective measures will be in accord with ongoing good laboratory practices and the overall Quality Assurance Program.

## SECTION 11 - PREVENTIVE MAINTENANCE

Preventive maintenance procedures will be carried out on all field equipment in accordance with the procedures outlined by the manufacturer's equipment manuals. Any field equipment used during this project that is not covered by the standard operating procedures will have a specific maintenance instruction sheet prepared for it.

## SECTION 12 - DATA ASSESSMENT PROCEDURES

The O'Brien & Gere laboratories QA/QC group leader will be responsible for assessing the quality of the data generated. His assessment will be based upon instrument tuning criteria, surrogate recoveries, matrix spikes, duplicate analysis and reagent and field blank integrity. *not identified*

The QA/QC group leader will advise the *QAM* of any data which he believes should be rated as "unacceptable" or "preliminary" along with recommendations for corrective action, if deemed necessary.

Tentatively identified compounds (TIC's) will be brought to the attention of the Project Manager (PM) who has the responsibility of deciding whether to require additional verification or discard the data. *?*

The Quality Assurance Manager (QAM) has the responsibility of assessing the quality of the data generated by outside contract laboratories. The QAM will review both the analytical data and QA/QC reports and will report any inconsistencies to the PM along with recommendations concerning the acceptability of the data.

Finally, all analytical data will be submitted to and assessed by the FWS in accordance with their standard procedures.

*Combine with Section 8.*

*Use Page II data assessment proc.*

*What are these?*

*TIC DP data only.*

*no*



### SECTION 13 - CORRECTIVE ACTION PROCEDURES

Corrective action procedures that might be implemented from audit results or upon detection of data unacceptability are developed on a case-by-case basis.

The actions may include:

- Reanalyzing samples if holding time requirements have not been exceeded.
- Altering field or handling procedures.
- Resampling.
- Using a different batch of sample containers.
- Recommending an audit of laboratory procedures.
- Accepting data with knowledged level of uncertainty.
- Discard data.

Further guidance to corrective actions is outlined in Attachment 4 to this QAPP. The O'Brien & Gere Project Manager is responsible for initiating the corrective action. The Regional Resource Contaminants Assessment Coordinator is responsible for approving the corrective action.

#### SECTION 14 - QUALITY ASSURANCE REPORTS

For this project, no separate report is anticipated to describe the performance of the data measurement systems or the data quality. Discussions of quality assurance problems and corrective actions taken will be included in the project monthly progress reports. The final RI report and the final FS report will contain separate QA sections that summarize data quality information collected during the project.

*I think that results of quarterly  
PE sample results is reported  
to F&W & U.S. EPA,*

# Attachments



**O'BRIEN & GERE**

ATTACHMENT 1

SAMPLING AND ANALYSIS SCHEDULE

- a. Key
- b. Phase I Listing
- c. Phase II Listing

CRAB ORCHARD NATIONAL WILDLIFE REFUGE  
SAMPLING & ANALYSIS SCHEDULE

KEY

<u>Col. No.</u>		
1.	ID1 -	Site Number
2.	ID2 -	Sequential number at a given site
3.	ID3 -	Sample Matrix: soil-1; water-2; sediment-3; fish-4; turtles-5; crayfish-6
4.	ID4 -	Analysis set: A-1; B-2; C-3; D-4; E-5; F-6; G-7; H-8 (see parameter list for analysis sets)
5.	MATRIX -	soil, water, etc, (See ID3)
6.	Name -	Description of sampling locations
7.	Type -	Type of sample collection - surface, grab, composite, core, etc.
8.	Depth -	Depth at which sample is collected
9.	Analysis Set -	See Parameter List for analysis sets
10-12.	Rationale -	For selection of sampling depth, location and intervals. See Attachment 2 for explanations.
13.	Lab No. -	Number used by OB&G Laboratory System
14.	Replicate -	Shows if replicates are collected for FWS, duplicates or spikes
15.	Sample Coll. Date -	Date of Sampling - '***' indicates sample collected; '---' indicates sample not collected
16.	Dupl/Spike - Numbers	Lab No. for corresponding duplicate or spike sample.

## PHASE 1 LISTING OF SAMPLES SCHEDULED

## CRAB ORCHARD NATIONAL WILDLIFE REFUGE

## SAMPLING AND ANALYSIS SCHEDULE

Revised March 17, 1986

## PHASE I - ALL SAMPLES

ID1	ID2	ID3	ID4	MATRIX	NAME	TYPE	DEPTH	ANAL	DEPTH	LOCA	INTRVL	SAMP	LAB	REPLICATE	SAMPLE	DUPL./SPIKE	NOTES
ST	N	MAT	A.S					SET		TION	% NO.	NOS.	NO	LAB FWS	COLL.	DATE	NUMBERS
!....(RATIONALE)....!																	
-----																	
GROUP: #1 SITE: 3:AREA 11 SOUTH LANDFILL																	
-----																	
3-	1-	1-	1	SOIL	NORTH BANK	COMP. 6 GRABS	0-1 FT	A	I	P	Y	1	9401	FWS	#1*	8/14/85	
3-	2-	1-	1	SOIL	SOUTH BANK	COMP. 6 GRABS	0-1 FT	A	I	P	Y	2	9402	DUPL	#1*	8/14/85	19221
3-	2-	1-	6	SOIL	SOUTH BANK	COMP. 6 GRABS	0-1 FT	F	I	P	Y	572	9257	DUPL	#1*	11/19/85	19222
3-	3-	1-	1	SOIL	EAST MOUND	COMP. 4 GRABS	0-1 FT	A	I	P	W	3	9403	SPKE	#1*	8/14/85	19266
3-	4-	3-	4	SEDIMENT	MARSH	COMP. 10 GRABS	0-1 FT	D	K	R,S	Y	4	9404		#1*	8/14/85	
3-	5-	3-	1	SEDIMENT	LOWER STREAM	COMP. 10 GRABS	0-1 FT	A	K	P,R,S	Y	5	9405		#1*	8/14/85	
-----																	
GROUP: #1 SITE: 4:AREA 11 NORTH LANDFILL																	
-----																	
4-	1-	1-	4	SOIL	BARE PATCHES	COMP. 6 GRABS	0-1 FT	D	I	P,U	Y	6	9406		#1*	8/13/85	
4-	2-	3-	1	SEDIMENT	SWAMPY SED.	COMP. 6 GRABS	0-1 FT	A	K	R	X	7	9407		#1*	8/13/85	
4-	2-	3-	6	SEDIMENT	SWAMPY SED.	COMP. 6 GRABS	0-1 FT	F	K	R	X	573	9258	FWS	#1*	11/19/85	
-----																	
GROUP: #1 SITE: 5:AREA 11 ACID POND																	
-----																	
5-	1-	2-	1	WATER	POND WATER	COMP. 4 GRABS	SURFACE	A	N	R	Y	8	9408		#1*	8/13/85	
5-	2-	1-	1	SOIL	DEAD TREE AREA	COMP. 4 GRABS	0-1 FT	A	K	P,R,U	Y	9	9409	FWS	#1*	8/13/85	
5-	3-	3-	1	SEDIMENT	POND SED.	COMP. 4 GRABS	0-1 FT	A	K	R	Y	10	9410		#1*	8/13/85	
5-	3-	3-	6	SEDIMENT	POND SED.	COMP. 4 GRABS	0-1 FT	F	K	R	Y	574	9259		#1*	11/19/85	

41 - GROUP: #2 SITE: 7A:D AREA NORTH LAWN  
 42 - -----  
 43 -  
 44 - 7A- 1- 1- 1 SOIL LOW SPOTS-SURF COMP. 8 GRABS SURFACE A+OVA I U X 11 9411 \*1# 8/17/85  
 45 - 7A- 2- 1- 1 SOIL LOW SPOTS-1 FT COMP. 8 GRABS 6-12 INCHES A+OVA M U X 12 9412 \*1# 8/17/85  
 46 - 7A- 3- 1- 1 SOIL LOW SPOTS-2 FT COMP. 8 GRABS 1-2 FEET A+OVA M U X 13 9413 FWS \*1# 8/17/85  
 47 - 7A- 4- 1- 1 SOIL LOW SPOTS-3 FT COMP. 8 GRABS 2-3 FEET A+OVA M U X 14 9414 \*1# 8/17/85  
 48 - 7A- 5- 1- 1 SOIL TRANSECT A-SURF COMP. 3 GRABS SURFACE A+OVA J Q X 15 9415 \*1# 8/17/85  
 49 - 7A- 6- 1- 1 SOIL TRANSECT A-1FT COMP. 3 GRABS 6-12 INCHES A+OVA M Q X 16 9416 \*1# 8/17/85  
 50 - 7A- 7- 1- 1 SOIL TRANSECT A-2FT COMP. 3 GRABS 1-2 FEET A+OVA M Q X 17 9417 DUPL \*1# 8/17/85 19226  
 51 - 7A- 8- 1- 1 SOIL TRANSECT A-3FT COMP. 3 GRABS 2-3 FEET A+OVA M Q X 18 9418 \*1# 8/17/85  
 52 - 7A- 9- 1- 1 SOIL TRANSECT B-SURF COMP. 3 GRABS SURFACE A+OVA J Q X 19 9419 \*1# 8/17/85  
 53 - 7A- 9- 1- 6 SOIL TRANSECT B-SURF COMP. 3 GRABS SURFACE F M Q X 575 9260 \*1# 11/19/85  
 54 - 7A- 10- 1- 1 SOIL TRANSECT B-1FT COMP. 3 GRABS 6-12 INCHES A+OVA M Q X 20 9420 \*1# 8/17/85  
 55 - 7A- 11- 1- 1 SOIL TRANSECT B-2FT COMP. 3 GRABS 1-2 FEET A+OVA M Q X 21 9421 \*1# 8/17/85  
 56 - 7A- 12- 1- 1 SOIL TRANSECT B-3FT COMP. 3 GRABS 2-3 FEET A+OVA M Q X 22 9422 \*1# 8/17/85  
 57 - 7A- 13- 1- 1 SOIL TRANSECT C-SURF COMP. 3 GRABS SURFACE A+OVA J Q X 23 9423 \*1# 8/17/85  
 58 - 7A- 14- 1- 1 SOIL TRANSECT C-1FT COMP. 3 GRABS 6-12 INCHES A+OVA M Q X 24 9424 \*1# 8/17/85  
 59 - 7A- 15- 1- 1 SOIL TRANSECT C-2FT COMP. 3 GRABS 1-2 FEET A+OVA M Q X 25 9425 \*1# 8/17/85  
 60 - 7A- 16- 1- 1 SOIL TRANSECT C-3FT COMP. 3 GRABS 2-3 FEET A+OVA M Q X 26 9426 \*1# 8/17/85

61 -  
 62 -  
 63 -  
 64 - GROUP: #2 SITE: 11A:P AREA NORTH  
 65 - -----  
 66 -  
 67 - 11A- 1- 3- 1 SEDIMENT WEST SWALE COMP. 3 GRABS 0-1 FT A J,K Q,R X 27 9427 SPKE \*1# 8/16/85 19282  
 68 - 11A- 2- 3- 1 SEDIMENT EAST SWALE COMP. 7 GRABS 0-1 FT A J,K Q,R X 28 9428 FWS \*1# 8/16/85  
 69 - 11A- 3- 3- 1 SEDIMENT NORTH SWALE 1 COMP. 6 GRABS 0-1 FT A J,K Q,R X 29 9429 DUPL \*1# 8/16/85 19254  
 70 - 11A- 3- 3- 6 SEDIMENT NORTH SWALE 1 COMP. 6 GRABS 0-1 FT F J,K Q,R X 576 9261 DUPL \*1# 11/18/85 19225  
 71 - 11A- 4- 3- 1 SEDIMENT NORTH SWALE 2 COMP. 3 GRABS 0-1 FT A J,K Q,R X 30 9430 \*1# 8/16/85  
 72 - 11A- 5- 1- 1 SOIL LOADING DOCK COMP. 3 GRABS 0-1 FT A J Q W 31 9431 \*1# 8/16/85  
 73 - 11A- 6- 1- 1 SOIL NORTH DOOR COMP. 2 GRABS 0-1 FT A J Q W 32 9432 \*1# 8/16/85  
 74 - 11A- 7- 1- 1 SOIL EAST LOAD AREA COMP. 3 GRABS 0-1 FT A J Q W 33 9433 \*1# 8/16/85  
 75 - 11A- 8- 1- 1 SOIL STEAMHOUSE DOOR COMP. 2 GRABS 0-1 FT A J Q W 34 9434 \*1# 8/16/85

76 -  
 77 -  
 78 -  
 79 - GROUP: #2 SITE: 7:D AREA SOUTHEAST DRAINAGE  
 80 - -----



81 -  
82 - 7- 1- 2- 1 WATER D-SE WATER COMP. 4 GRABS SURFACE A N R W 35 19209 \*1\* 8/16/85  
83 - 7- 2- 3- 1 SEDIMENT D-SE SEDIMENT COMP. 4 GRABS 0-1 FT A K R W 36 19210 DUPL \*1\* 8/16/85 19255  
84 -  
85 -  
86 -  
87 - -----  
88 - GROUP: #2 SITE: 8:D AREA SOUTHWEST DRAINAGE  
89 - -----  
90 - 8- 1- 2- 1 WATER D-SW WATER COMP. 2 GRABS SURFACE A N R W 37 3258 \*1\* 7/25/85  
91 - 8- 2- 3- 1 SEDIMENT D-SW SEDIMENT COMP. 4 GRABS 0-1 FT A K R W 38 3386 \*1\* 7/25/85  
92 -  
93 -  
94 -  
95 - -----  
96 - GROUP: #2 SITE: 9:D AREA NORTHWEST DRAINAGE  
97 - -----  
98 - 9- 1- 2- 1 WATER P-NW WATER COMP. 4 GRABS SURFACE A N R W 39 3257 \*1\* 7/25/85  
99 - 9- 2- 3- 1 SEDIMENT P-NW SEDIMENT COMP. 4 GRABS 0-1 FT A K R W 40 3385 \*1\* 7/25/85  
100 -  
101 -  
102 -  
103 - -----  
104 - GROUP: #2 SITE: 10:WATERWORKS NORTH DRAINAGE  
105 - -----  
106 - 10- 1- 2- 1 WATER WW-N WATER COMP. 4 GRABS SURFACE A N R,S W 41 3250 \*1\* 7/25/85  
107 - 10- 2- 3- 4 SEDIMENT WW-N SEDIMENT COMP. 4 GRABS 0-1 FT D K R,S W 42 42 FWS \*1\* 7/25/85  
108 - 10- 2- 3- 7 SEDIMENT WW-N SEDIMENT COMP. 4 GRABS 0-1 FT G K R,S W 577 9262 DUPL \*1\* 11/19/85 19259  
109 -  
110 -  
111 -  
112 - -----  
113 - GROUP: #2 SITE: 11:P AREA SOUTHEAST DRAINAGE  
114 - -----  
115 - 11- 1- 2- 1 WATER P-SE WATER COMP. 4 GRABS SURFACE A N R W 43 3247 \*1\* 7/25/85  
116 - 11- 2- 3- 1 SEDIMENT P-SE SEDIMENT COMP. 4 GRABS 0-1 FT A K R W 44 44 \*1\* 7/25/85  
117 - 11- 2- 3- 6 SEDIMENT P-SE SEDIMENT COMP. 4 GRABS 0-1 FT F K R W 578 9263 \*1\* 11/18/85  
118 -  
119 -  
120 - -----

## GROUP: #2 SITE: 20:D AREA SOUTH

121 -  
 122 -  
 123 -  
 124 - 20- 2- 3- 1 SEDIMENT D SOUTH COMP. 4 GRABS 0-1 FT A K R X 46 3389 #1# 7/25/85  
 125 - 20- 2- 3- 6 SEDIMENT D SOUTH COMP. 4 GRABS 0-1 FT F K R X 579 9264 #1# 11/18/85  
 126 -  
 127 -  
 128 -

## GROUP: #3 SITE: 12:AREA 14 LANDFILL

129 -  
 130 -  
 131 -  
 132 - 12- 2- 3- 1 SEDIMENT DRAINAGE CHANNEL COMP. 4 GRABS 0-1 FT A I,K P,R W 48 3387 DUPL #1# 7/25/85 9255  
 133 - 12- 2- 3- 7 SEDIMENT DRAINAGE CHANNEL COMP. 4 GRABS 0-1 FT G I,K P,R W 580 9265 #1# 11/18/85  
 134 - 12- 3- 1- 4 SOIL BLACK RESIDUE COMP. 4 GRABS 0-1 FT D I P,U W 49 9385 FWS #1# 8/14/85  
 135 -  
 136 -  
 137 -

## GROUP: #3 SITE: 13:AREA 14 CHANGE HOUSE SITE

138 -  
 139 -  
 140 -  
 141 - 13- 1- 1- 1 SOIL TRANSECT 1 COMP. 10 GRABS 0-1 FT A J Q Y 50 9386 #1# 8/15/85 CLOSEST TO RD.  
 142 - 13- 2- 1- 1 SOIL TRANSECT 2 COMP. 10 GRABS 0-1 FT A J Q Y 51 9387 FWS #1# 8/15/85  
 143 - 13- 3- 1- 1 SOIL TRANSECT 3 COMP. 10 GRABS 0-1 FT A J Q Y 52 9388 #1# 8/15/85  
 144 - 13- 4- 1- 1 SOIL TRANSECT 4 COMP. 10 GRABS 0-1 FT A J Q Y 53 9389 #1# 8/15/85  
 145 - 13- 5- 1- 1 SOIL TRANSECT 5 COMP. 10 GRABS 0-1 FT A J Q Y 54 9390 #1# 8/15/85  
 146 - 13- 6- 1- 1 SOIL TRANSECT 6 COMP. 10 GRABS 0-1 FT A J Q Y 55 9391 #1# 8/15/85  
 147 -  
 148 -  
 149 -

## GROUP: #3 SITE: 14:AREA 14 SOLVENT STORAGE

150 -  
 151 -  
 152 -  
 153 - 14- 1- 2- 1 WATER DITCH NORTH COMP. 6 GRABS SURFACE A N Q,R Y 56 19301 #1# 7/25/85  
 154 - 14- 2- 3- 1 SEDIMENT DITCH NORTH COMP. 6 GRABS 0-1 FT A K Q,R Y 57 19302 FWS #1# 7/25/85  
 155 - 14- 3- 2- 1 WATER DITCH SOUTH COMP. 6 GRABS SURFACE A N Q,R Y 58 19303 #1# 7/25/85  
 156 - 14- 4- 3- 1 SEDIMENT DITCH SOUTH COMP. 6 GRABS 0-1 FT A K Q,R Y 59 19304 #1# 7/25/85  
 157 - 14- 4- 3- 6 SEDIMENT DITCH SOUTH COMP. 6 GRABS 0-1 FT F K Q,R Y 581 9266 #1# 11/18/85  
 158 -  
 159 -  
 160 -



201 -	17-	5-	1-	1	SOIL	SOIL GRID 5	COMP. 5 GRABS	0-1 FT	A	I	P	X	84	9445		*1*	8/16/85	
202 -	17-	6-	1-	4	SOIL	BARE PATCH 1	COMP. 2 GRABS	0-1 FT	D	I	P,U	W	85	9446		*1*	8/16/85	
203 -	17-	6-	1-	7	SOIL	BARE PATCH 1	COMP. 2 GRABS	0-1 FT	G	I	P,U	W	586	9271		*1*	11/18/85	
204 -	17-	7-	1-	1	SOIL	BARE PATCH 2	COMP. 2 GRABS	SURFACE	A	I	P,U	W	86	9447	FWS	*1*	8/16/85	
205 -	17-	8-	2-	17	WATER	WELL 17-1	SINGLE SAMPLE	BAILER	Q	-	-	-	87	9448		.1.		
206 -	17-	9-	2-	17	WATER	WELL 17-2	SINGLE SAMPLE	BAILER	Q	-	-	-	88	9449		.1.		
207 -	17-	10-	2-	17	WATER	WELL 17-3	SINGLE SAMPLE	BAILER	Q	-	-	-	89	9450 DUPL		.1.		19219
208 -	17-	11-	2-	17	WATER	WELL 17-4	SINGLE SAMPLE	BAILER	Q	-	-	-	90	9451		.1.		
209 -	17-	12-	2-	1	WATER	POND NO.1	SINGLE SAMPLE	SURFACE	A	K,N	R	W	91	3248		*1*	7/25/85	
210 -	17-	13-	2-	1	WATER	POND NO.2	SINGLE SAMPLE	SURFACE	A	K,N	R	W	92	3249		*1*	7/25/85	

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 GROUP: #6 SITE: 18:AREA 13 LOADING PLATFORM  
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217 -	18-	1-	1-	1	SOIL	LOADING DOCK N	COMP. 20 GRABS	0-1 FT	A	J	Q	Y	93	9452	FWS	*1*	8/15/85	
218 -	18-	2-	1-	1	SOIL	LOADING DOCK S	COMP. 20 GRABS	0-1 FT	A	J	Q	Y	94	9453 DUPL		*1*	8/15/85	19223
219 -	18-	3-	1-	1	SOIL	LOADING DOCK E	COMP. 2 GRABS	0-1 FT	A	J	Q	W	95	9454		*1*	8/15/85	
220 -	18-	4-	1-	1	SOIL	LOADING DOCK W	COMP. 2 GRABS	0-1 FT	A	I	P,Q	W	96	9455		*1*	8/15/85	
221 -	18-	4-	1-	6	SOIL	LOADING DOCK W	COMP. 2 GRABS	0-1 FT	F	I	P,Q	W	587	9272		*1*	11/19/85	

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 GROUP: #6 SITE: 19:AREA 13 BUNKER 1-3  
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228 -	19-	1-	1-	1	SOIL	SOIL GRID NE	COMP. 14 GRABS	0-1 FT	A	J	Q	Y	97	9456		*1*	8/16/85	
229 -	19-	2-	1-	1	SOIL	SOIL GRID SE	COMP. 14 GRABS	0-1 FT	A	J	Q	Y	98	9457		*1*	8/16/85	
230 -	19-	3-	1-	1	SOIL	SOIL GRID NW	COMP. 14 GRABS	0-1 FT	A	J	Q	Y	99	9458		*1*	8/16/85	
231 -	19-	3-	1-	6	SOIL	SOIL GRID NW	COMP. 14 GRABS	0-1 FT	F	J	Q	Y	588	9273		*1*	11/19/85	
232 -	19-	4-	1-	1	SOIL	SOIL GRID FRONT	COMP. 10 GRABS	0-1 FT	A	J	Q	Y	100	9459 SPKE		*1*	8/16/85	19268
233 -	19-	5-	1-	1	SOIL	BR. PATCH TRANSECT	COMP. 3 GRABS	0-1 FT	A	J	Q,U	X	101	9460	FWS	*1*	8/16/85	

-----  
 GROUP: #6 SITE: 30:MUNITIONS CONTROL SITE  
 -----

240 -	30-	1-	1-	4	SOIL	MUNITION CONTROL	SINGLE SAMPLE	SURFACE	D	-	T	W	102	9461 DUPL FWS		*1*	8/16/85	19289 BUNKER 1-11
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241 -	30-	1-	1-	7	SOIL	MUNITION CONTROL	SINGLE SAMPLE	SURFACE	G	-	T	W	589	9274	FWS	*1*	11/19/85	
242 -	30-	2-	2-	9	WATER	MUNITION CONTROL	SINGLE SAMPLE	BAILER	I	-	T	W	103	9462		.1.		
243 -																		
244 -																		
245 -																		
246 -																		
247 -																		
248 -																		
249 -	21-	1-	1-	1	SOIL	TRANSECT 1	COMP. 6 GRABS	0-1 FT	A	J	Q	Y	104	9463 DUSP		*1*	8/14/85	19224 1st, SPKE 14138
250 -	21-	1-	1-	6	SOIL	TRANSECT 1	COMP. 6 GRABS	0-1 FT	F	J	Q	Y	590	9275		*1*	11/19/85	
251 -	21-	2-	1-	1	SOIL	TRANSECT 2	COMP. 6 GRABS	0-1 FT	A	J	Q	Y	105	9464		*1*	8/14/85	4th FROM RD.
252 -	21-	3-	1-	1	SOIL	TRANSECT 3	COMP. 6 GRABS	0-1 FT	A	J	Q	Y	106	9465		*1*	8/14/85	6th FROM RD.
253 -	21-	4-	1-	1	SOIL	TRANSECT 4	COMP. 6 GRABS	0-1 FT	A	J	Q	Y	107	9466		*1*	8/14/85	8th FROM RD.
254 -																		
255 -																		
256 -																		
257 -																		
258 -																		
259 -																		
260 -	22-	1-	2-	1	WATER	POOL WATER	SINGLE GRAB	SURFACE	A	K,N	P	W	108	3256		*1*	7/25/85	
261 -	22-	2-	3-	1	SEDIMENT	STREAM SEDIMENTS	COMP. 2 GRABS	0-1 FT	A	K	Q,R	W	109	3384	FWS	*1*	7/25/85	
262 -	22-	2-	3-	6	SEDIMENT	STREAM SEDIMENTS	COMP. 2 GRABS	0-1 FT	F	K	Q,R	W	591	9276		*1*	12/05/85	
263 -																		
264 -																		
265 -																		
266 -																		
267 -																		
268 -																		
269 -	24-	1-	2-	1	WATER	PEPSI-WEST	COMP. 3 GRABS	SURFACE	A	K,N	R	W	110	3254		*1*	7/25/85	
270 -	24-	2-	3-	1	SEDIMENT	PEPSI-WEST	COMP. 3 GRABS	0-1 FT	A	K	R	W	111	2711		*1*	7/25/85	
271 -	24-	2-	3-	6	SEDIMENT	PEPSI-WEST	COMP. 3 GRABS	0-1 FT	F	K	R	W	593	9278 SPKE		*1*	12/05/85	19262
272 -																		
273 -																		
274 -																		
275 -																		
276 -																		
277 -																		
278 -	25-	1-	2-	1	WATER	COC DOWNSTREAM	COMP. 3 GRABS	SURFACE	A	K,N	R	W	112	3243		*1*	7/25/85	
279 -	25-	2-	3-	4	SEDIMENT	COC DOWNSTREAM	COMP. 3 GRABS	0-1 FT	D	K	R	W	113	3388		*1*	7/25/85	
280 -	25-	3-	3-	7	SEDIMENT	COC DOWNSTREAM	COMP. 3 GRABS	0-1 FT	G	K	R	W	594	9279		*1*	12/05/85	

281 -	25-	3-	2-	1	WATER	COC UPSTREAM	COMP. 3 GRABS	SURFACE	A	-	T	W	114	9467 DUSP	#1*	8/13/85	19215	SPKE 85576
282 -	25-	4-	3-	1	SEDIMENT	COC UPSTREAM	COMP. 3 GRABS	0-1 FT	A	-	T	W	115	9468 DUPL	#1*	8/13/85	19256	
283 -	25-	5-	2-	1	WATER	LF POND	COMP. 3 GRABS	SURFACE	A	K,N	R	W	116	9469	#1*	8/13/85		
284 -	25-	6-	3-	1	SEDIMENT	LF POND	COMP. 3 GRABS	0-1 FT	A	K	R	W	117	9470 FWS	#1*	8/13/85		
285 -																		
286 -																		
287 -																		
288 -																		
289 -																		
290 -																		
291 -	26-	1-	2-	1	WATER	COC AT S. CARBON	COMP. 3 GRABS	SURFACE	A	K,N	R	W	118	3244	#1*	7/25/85		
292 -	26-	2-	3-	1	SEDIMENT	COC AT S. CARBON	COMP. 3 GRABS	0-1 FT	A	K	R	W	119	3391	#1*	7/25/85		
293 -	26-	3-	2-	1	WATER	COC AT COURT ST.	COMP. 3 GRABS	SURFACE	A	K,N	R	W	120	9471	#1*	8/13/85		
294 -	26-	4-	3-	1	SEDIMENT	COC AT COURT ST.	COMP. 3 GRABS	0-1 FT	A	K	R	W	121	9472 FWS	#1*	8/13/85		
295 -																		
296 -																		
297 -																		
298 -																		
299 -																		
300 -																		
301 -	27-	1-	2-	1	WATER	COC AT CHAMNESS	COMP. 3 GRABS	SURFACE	A	K,N	R,S	W	122	3245	#1*	7/25/85		
302 -	27-	2-	3-	4	SEDIMENT	COC AT CHAMNESS	COMP. 3 GRABS	0-1 FT	D	K	R,S	W	123	3390	#1*	7/25/85		
303 -																		
304 -																		
305 -																		
306 -																		
307 -																		
308 -																		
309 -	28-	1-	1-	4	SOIL	MAIN GULLY	COMP. 8 GRABS	0-1 FT	D	K	R,S	X	124	9473 DUPL	#1*	8/14/85	19291	
310 -	28-	2-	1-	1	SOIL	TRANS. GULLY	COMP. 6 GRABS	0-1 FT	A	I,K	P,R	X	125	9474	#1*	8/14/85		
311 -	28-	2-	1-	7	SOIL	TRANS. GULLY	COMP. 6 GRABS	0-1 FT	G	I,K	P,R	X	596	9281 SPKE	#1*	11/19/85	19263	
312 -	28-	3-	1-	1	SOIL	SOIL GRID 1	COMP. 6 GRABS	0-1 FT	A	J	Q	X	126	9475 FWS	#1*	8/14/85		
313 -	28-	4-	1-	1	SOIL	SOIL GRID 2	COMP. 6 GRABS	0-1 FT	A	J	Q	X	127	9476	#1*	8/14/85		
314 -	28-	5-	1-	1	SOIL	SOIL GRID 3	COMP. 6 GRABS	0-1 FT	A	J	Q	X	128	9477	#1*	8/14/85		
315 -	28-	6-	1-	1	SOIL	SOIL GRID 4	COMP. 6 GRABS	0-1 FT	A	J	Q	X	129	9478	#1*	8/14/85		
316 -	28-	7-	2-	19	WATER	WELL 28-1	SINGLE GRAB	BAILER	S	-	V	W	130	9479	.1.			
317 -	28-	8-	2-	19	WATER	WELL 28-2	SINGLE GRAB	BAILER	S	-	V	W	131	9480	.1.			
318 -	28-	9-	1-	1	SOIL	SOUTH END OF DITCH	GRAB	0-1 FT	A	I,K	P,R	W	132	9481 SPKE	#1*	8/14/85	19269	
319 -	28-	10-	1-	1	SOIL	NORTH END OF DITCH	GRAB	0-1 FT	A	I,K	P,R	W	133	9482	#1*	8/14/85		
320 -	28-	11-	1-	1	SOIL	NORTH OF 28-4	GRAB	0-1 FT	A	I	P	W	134	9483	#1*	8/14/85		

321 -	28- 12-	1- 1	SOIL	NORTHWEST OF 28-3	GRAB	0-1 FT	A	I	P	W	135	9484		*1*	8/14/85	
322 -	28- 13-	1- 1	SOIL	N. FIELD-OLD 28-9	GRAB	0-1 FT	A	I	P	W	136	9485		*1*	8/14/85	
323 -	28- 14-	1- 1	SOIL	GULLY	GRAB	0-1 FT	A	I,K	P,R	W	137	9486		*1*	8/14/85	
324 -																
325 -																
326 -																
327 -																
328 -																
329 -																
330 -	29- 1-	1- 1	SOIL	EAST FACE 1	COMP. 12 GRABS	0&1 FT	A	I	P	X	138	9487 SPKE		*1*	8/13/85	19288
331 -	29- 2-	1- 1	SOIL	EAST FACE 2	COMP. 12 GRABS	0&1 FT	A	I	P	X	139	9488		*1*	8/13/85	
332 -	29- 2-	1- 7	SOIL	EAST FACE 2	COMP. 12 GRABS	0&1 FT	G	I	P	X	597	9282		*1*	11/19/85	
333 -	29- 3-	1- 4	SOIL	EAST FACE 3	COMP. 12 GRABS	0&1 FT	D	I	P	X	140	9489		*1*	8/13/85	
334 -	29- 4-	1- 1	SOIL	EAST FACE 4	COMP. 12 GRABS	0&1 FT	A	I	P	X	141	9490		*1*	8/13/85	
335 -	29- 5-	1- 1	SOIL	NORTH FACE 1	COMP. 12 GRABS	0&1 FT	A	I	P	X	142	9491 DUPL		*1*	8/13/85	19287
336 -	29- 6-	1- 4	SOIL	NORTH FACE 2	COMP. 12 GRABS	0&1 FT	D	I	P	X	143	9492		*1*	8/13/85	
337 -	29- 7-	1- 1	SOIL	NORTH FACE 3	COMP. 12 GRABS	0&1 FT	A	I	P	X	144	9493 FWS		*1*	8/13/85	
338 -	29- 8-	2- 19	WATER	WELL 29-1	SINGLE GRAB	BAILER	S	-	T	W	145	9494		.1.		
339 -	29- 9-	2- 19	WATER	WELL 29-2	SINGLE GRAB	BAILER	S	-	V	W	146	9495		.1.		
340 -	29- 10-	2- 19	WATER	WELL 29-3	SINGLE GRAB	BAILER	S	-	V	W	147	9496		.1.		
341 -	29- 11-	2- 19	WATER	WELL 29-4	SINGLE GRAB	BAILER	S	-	V	W	148	9497		.1.		
342 -																
343 -																
344 -																
345 -																
346 -																
347 -																
348 -	32- 1-	1- 8	SOIL	SOIL GRID 1	COMP.@1'DEPTHS	0-12 FT	H	L,M	P	Z	149	9498		*1*	8/24/85	
349 -	32- 2-	1- 3	SOIL	SOIL GRID 1-0	TOP CORE COMP.	0-6 INCH	C	I	P	Z	150	9499		*1*	8/24/85	
350 -	32- 3-	1- 3	SOIL	SOIL GRID 1-1	MID CORE COMP.	6-6.5 FT	C	L,M	P	Z	151	9500 DUPL		*1*	8/24/85	19250
351 -	32- 4-	1- 3	SOIL	SOIL GRID 1-2	BOT CORE COMP.	11.5-12'	C	L,M	P	Z	152	10640		*1*	8/24/85	
352 -	32- 5-	1- 8	SOIL	SOIL GRID 2	COMP.@1'DEPTHS	0-12 FT	H	L,M	P	Z	153	10641		*1*	8/24/85	
353 -	32- 6-	1- 3	SOIL	SOIL GRID 2-0	TOP CORE COMP.	0-6 INCH	C	I	P	Z	154	10642		*1*	8/24/85	
354 -	32- 7-	1- 3	SOIL	SOIL GRID 2-1	MID CORE COMP.	6-6.5 FT	C	L,M	P	Z	155	10643		*1*	8/24/85	
355 -	32- 8-	1- 3	SOIL	SOIL GRID 2-2	BOT CORE COMP.	11.5-12'	C	L,M	P	Z	156	10644		*1*	8/24/85	
356 -	32- 9-	1- 8	SOIL	SOIL GRID 3	COMP.@1'DEPTHS	0-12 FT	H	L,M	P	Z	157	10645		*1*	8/21/85	
357 -	32- 10-	1- 3	SOIL	SOIL GRID 3-0	TOP CORE COMP.	0-6 INCH	C	I	P	Z	158	10646		*1*	8/21/85	
358 -	32- 11-	1- 3	SOIL	SOIL GRID 3-1	MID CORE COMP.	6-6.5 FT	C	L,M	P	Z	159	10647		*1*	8/21/85	
359 -	32- 12-	1- 3	SOIL	SOIL GRID 3-2	BOT CORE COMP.	11.5-12'	C	L,M	P	Z	160	10648		*1*	8/21/85	
360 -	32- 13-	1- 8	SOIL	SOIL GRID 4	COMP.@1'DEPTHS	0-12 FT	H	L,M	P	Z	161	10649		*1*	8/21/85	

361 -	32- 14-	1- 3	SOIL	SOIL GRID 4-0	TOP CORE COMP.	0-6 INCH	C	I	P	Z	162	10650		*1*	8/21/85	
362 -	32- 15-	1- 3	SOIL	SOIL GRID 4-1	MID CORE COMP.	6-6.5 FT	C	L,M	P	Z	163	10651		*1*	8/21/85	
363 -	32- 16-	1- 3	SOIL	SOIL GRID 4-2	BOT CORE COMP.	11.5-12'	C	L,M	P	Z	164	10652		*1*	8/21/85	
364 -	32- 17-	1- 8	SOIL	SOIL GRID 5	COMP.#1'DEPTHS	0-12 FT	H	L,M	P	Z	165	10653		*1*	8/22/85	
365 -	32- 18-	1- 3	SOIL	SOIL GRID 5-0	TOP CORE COMP.	0-6 INCH	C	I	P	Z	166	10654	DUPL	*1*	8/22/85	19249
366 -	32- 19-	1- 3	SOIL	SOIL GRID 5-1	MID CORE COMP.	6-6.5 FT	C	L,M	P	Z	167	10655		*1*	8/22/85	
367 -	32- 20-	1- 3	SOIL	SOIL GRID 5-2	BOT CORE COMP.	11.5-12'	C	L,M	P	Z	168	10656		*1*	8/22/85	
368 -	32- 21-	1- 8	SOIL	SOIL GRID 6	COMP.#1'DEPTHS	0-12 FT	H	L,M	P	Z	169	10657		*1*	8/22/85	
369 -	32- 22-	1- 3	SOIL	SOIL GRID 6-0	TOP CORE COMP.	0-6 INCH	C	I	P	Z	170	10658	FWS	*1*	8/22/85	
370 -	32- 23-	1- 3	SOIL	SOIL GRID 6-1	MID CORE COMP.	6-6.5 FT	C	L,M	P	Z	171	10659		*1*	8/22/85	
371 -	32- 24-	1- 3	SOIL	SOIL GRID 6-2	BOT CORE COMP.	11.5-12'	C	L,M	P	Z	172	10660	DUPL	*1*	8/22/85	19251
372 -	32- 25-	1- 8	SOIL	SOIL GRID 7	COMP.#1'DEPTHS	0-12 FT	H	L,M	P	Z	173	10661	DUPL	*1*	8/22/85	19252
373 -	32- 26-	1- 3	SOIL	SOIL GRID 7-0	TOP CORE COMP.	0-6 INCH	C	I	P	Z	174	10662		*1*	8/22/85	
374 -	32- 27-	1- 3	SOIL	SOIL GRID 7-1	MID CORE COMP.	6-6.5 FT	C	L,M	P	Z	175	10663		*1*	8/22/85	
375 -	32- 28-	1- 3	SOIL	SOIL GRID 7-2	BOT CORE COMP.	11.5-12'	C	L,M	P	Z	176	10664		*1*	8/22/85	
376 -	32- 29-	1- 8	SOIL	SOIL GRID 8	COMP.#1'DEPTHS	0-12 FT	H	L,M	P	Z	177	10665	SPKE	*1*	8/23/85	19281
377 -	32- 30-	1- 3	SOIL	SOIL GRID 8-0	TOP CORE COMP.	0-6 INCH	C	I	P	Z	178	10666	SPKE	*1*	8/23/85	19280
378 -	32- 31-	1- 3	SOIL	SOIL GRID 8-1	MID CORE COMP.	6-6.5 FT	C	L,M	P	Z	179	10667	FWS	*1*	8/23/85	
379 -	32- 32-	1- 3	SOIL	SOIL GRID 8-2	BOT CORE COMP.	11.5-12'	C	L,M	P	Z	180	10668		*1*	8/23/85	
380 -	32- 33-	1- 8	SOIL	SOIL GRID 9	COMP.#1'DEPTHS	0-12 FT	H	L,M	P	Z	181	10669	FWS	*1*	8/23/85	
381 -	32- 34-	1- 3	SOIL	SOIL GRID 9-0	TOP CORE COMP.	0-6 INCH	C	I	P	Z	182	10670	FWS	*1*	8/23/85	
382 -	32- 35-	1- 3	SOIL	SOIL GRID 9-1	MID CORE COMP.	6-6.5 FT	C	L,M	P	Z	183	10671		*1*	8/23/85	
383 -	32- 36-	1- 3	SOIL	SOIL GRID 9-2	BOT CORE COMP.	11.5-12'	C	L,M	P	Z	184	10672		*1*	8/23/85	
384 -	32- 37-	1- 2	SOIL	NORTH TRANSECT 1	COMP.#3'INTRVL	SURFACE	B	K	R or T	Y	185	10673		*1*	8/19/85	
385 -	32- 38-	1- 2	SOIL	NORTH TRANSECT 1	COMP.#3'INTRVL	SURFACE	B	K	R or T	Y	186	10674		*1*	8/19/85	
386 -	32- 39-	1- 2	SOIL	EAST TRANSECT 1	COMP.#3'INTRVL	SURFACE	B	K	R or T	Y	187	10675		*1*	8/19/85	
387 -	32- 40-	1- 2	SOIL	EAST TRANSECT 2	COMP.#3'INTRVL	SURFACE	B	K	R or T	Y	188	10676		*1*	8/19/85	
388 -	32- 41-	1- 2	SOIL	SOUTH TRANSECT 1	COMP.#3'INTRVL	SURFACE	B	K	R or T	Y	189	10677		*1*	8/19/85	
389 -	32- 42-	1- 2	SOIL	SOUTH TRANSECT 2	COMP.#3'INTRVL	SURFACE	B	K	R or T	Y	190	10678		*1*	8/19/85	
390 -	32- 43-	3- 1	SEDIMENT	INT. CREEK 1-0	GRAB	SURFACE	A	J,K	Q,R	Z	191	10679		*1*	8/22/85	
391 -	32- 44-	3- 1	SEDIMENT	INT. CREEK 1-1	GRAB	3 FEET	A	J,K,L	Q,R	Z	192	10680		*1*	8/22/85	
392 -	32- 45-	3- 1	SEDIMENT	INT. CREEK 1-2	GRAB	6 FEET	A	J,K,L	Q,R	Z	193	10681		*1*	8/22/85	
393 -	32- 46-	3- 1	SEDIMENT	INT. CREEK 2-0	GRAB	SURFACE	A	J,K	Q,R	Z	194	10682	FWS	*1*	8/22/85	
394 -	32- 47-	3- 1	SEDIMENT	INT. CREEK 2-1	GRAB	3 FEET	A	J,K,L	Q,R	Z	195	10683		*1*	8/22/85	
395 -	32- 48-	3- 1	SEDIMENT	INT. CREEK 2-2	GRAB	6 FEET	A	J,K,L	Q,R	Z	196	10684		*1*	8/22/85	
396 -	32- 49-	3- 1	SEDIMENT	INT. CREEK 3-0	GRAB	SURFACE	A	J,K	Q,R	Z	197	10685	DUPL	*1*	8/22/85	19257
397 -	32- 50-	3- 1	SEDIMENT	INT. CREEK 3-1	GRAB	3 FEET	A	J,K,L	Q,R	Z	198	10686		*1*	8/22/85	
398 -	32- 51-	3- 1	SEDIMENT	INT. CREEK 3-2	GRAB	6 FEET	A	J,K,L	Q,R	Z	199	10687		*1*	8/22/85	
399 -	32- 52-	3- 1	SEDIMENT	INT. CREEK 4-0	GRAB	SURFACE	A	J,K	Q,R	Z	200	10688	FWS	*1*	8/23/85	
400 -	32- 53-	3- 1	SEDIMENT	INT. CREEK 4-1	GRAB	3 FEET	A	J,K,L	Q,R	Z	201	10689		*1*	8/23/85	



401 -	32- 54-	3-	1	SEDIMENT	INT. CREEK 4-2	GRAB	6 FEET	A	J,K,L, Q,R	Z	202	10690		*1*	8/23/85	
402 -	32- 55-	3-	1	SEDIMENT	INT. CREEK 5-0	GRAB	SURFACE	A	J,K Q,R	Z	203	10691	DUPL	*1*	8/23/85	19258
403 -	32- 56-	3-	1	SEDIMENT	INT. CREEK 5-1	GRAB	3 FEET	A	J,K,L, Q,R	Z	204	10692		*1*	8/23/85	
404 -	32- 57-	3-	1	SEDIMENT	INT. CREEK 5-2	GRAB	6 FEET	A	J,K,L, Q,R	Z	205	10693		*1*	8/23/85	
405 -	32- 58-	3-	4	SEDIMENT	INT. CREEK 6-0	GRAB	SURFACE	D	J,K Q,R	Z	206	10694	SPKE	*1*	8/23/85	19285
406 -	32- 59-	3-	4	SEDIMENT	INT. CREEK 6-1	GRAB	3 FEET	D	J,K,L, Q,R	Z	207	10695		*1*	8/23/85	
407 -	32- 60-	3-	4	SEDIMENT	INT. CREEK 6-2	GRAB	6 FEET	D	J,K,L, Q,R	Z	208	10696		*1*	8/23/85	
408 -	32- 61-	2-	9	WATER	WELL 1	SINGLE SAMPLE	BAILER	I	- T	W	209	10697		.1.		
409 -	32- 62-	2-	9	WATER	WELL 2	SINGLE SAMPLE	BAILER	I	- V	W	210	10698		.1.		
410 -	32- 63-	2-	9	WATER	WELL 3	SINGLE SAMPLE	BAILER	I	- V	W	211	10699		.1.		
411 -	32- 64-	1-	1	SOIL	YELLOW SPOT	SINGLE SAMPLE	SURFACE	A	I P	W	558	46701		*1*	8/26/85	
412 -	32- 65-	1-	2	SOIL	BEFORE CLEANING	SINGLE SAMPLE	SURFACE	B	K T	W	559	46702		*1*	8/26/85	
413 -	32- 66-	1-	2	SOIL	AFTER CLEANING	SINGLE SAMPLE	SURFACE	B	K T	W	560	46703		*1*	8/26/85	
414 -																
415 -																
416 -																
417 -																
418 -																
419 -																
420 -	33- 1-	1-	2	SOIL	LOC. 1 - I-1- 25	CORE VERTICAL	0-1 FOOT	B	J Q1	Z	212	10700	FWS	*1*	9/23/85	
421 -	33- 2-	1-	2	SOIL	LOC. 1 - I-1- 25	CORE VERTICAL	1-2 FEET	B	J,M Q1	Z	213	10701		*1*	9/23/85	
422 -	33- 3-	1-	2	SOIL	LOC. 1 - I-1- 25	CORE VERTICAL	2-3 FEET	B	J,M Q1	Z	214	10702		*1*	9/23/85	
423 -	33- 4-	1-	2	SOIL	LOC. 2 - I-1- 25	CORE VERTICAL	0-1 FOOT	B	J Q1	Z	215	10703	DUPL	*1*	9/23/85	19228
424 -	33- 5-	1-	2	SOIL	LOC. 2 - I-1- 25	CORE VERTICAL	1-2 FEET	B	J,M Q1	Z	216	10704		*1*	9/23/85	
425 -	33- 6-	1-	2	SOIL	LOC. 2 - I-1- 25	CORE VERTICAL	2-3 FEET	B	J,M Q1	Z	217	10705		*1*	9/23/85	
426 -	33- 7-	1-	2	SOIL	LOC. 3 - I-1- 25	CORE VERTICAL	0-1 FOOT	B	J Q1	Z	218	10706		*1*	9/23/85	
427 -	33- 8-	1-	2	SOIL	LOC. 3 - I-1- 25	CORE VERTICAL	1-2 FEET	B	J,M Q1	Z	219	10707		*1*	9/23/85	
428 -	33- 9-	1-	2	SOIL	LOC. 3 - I-1- 25	CORE VERTICAL	2-3 FEET	B	J,M Q1	Z	220	10708		*1*	9/23/85	
429 -	33- 10-	1-	2	SOIL	LOC. 4 - I-1- 25	CORE VERTICAL	0-1 FOOT	B	J Q1	Z	221	10709		*1*	9/23/85	
430 -	33- 11-	1-	2	SOIL	LOC. 4 - I-1- 25	CORE VERTICAL	1-2 FEET	B	J,M Q1	Z	222	10710		*1*	9/23/85	
431 -	33- 12-	1-	2	SOIL	LOC. 4 - I-1- 25	CORE VERTICAL	2-3 FEET	B	J,M Q1	Z	223	10711		*1*	9/23/85	
432 -	33- 13-	1-	2	SOIL	LOC. 5 - I-1- 25	CORE VERTICAL	0-1 FOOT	B	J Q1	Z	224	10712		*1*	9/23/85	
433 -	33- 14-	1-	2	SOIL	LOC. 5 - I-1- 25	CORE VERTICAL	1-2 FEET	B	J,M Q1	Z	225	10713		*1*	9/23/85	
434 -	33- 15-	1-	2	SOIL	LOC. 5 - I-1- 25	CORE VERTICAL	2-3 FEET	B	J,M Q1	Z	226	10714		*1*	9/23/85	
435 -	33- 16-	1-	2	SOIL	LOC. 6 - I-1- 25	CORE SURFACE	0-1 FOOT	B	J Q1	Z	227	10715	DUPL	*1*	9/23/85	19229
436 -	33- 17-	1-	2	SOIL	LOC. 7 - I-1- 25	CORE VERTICAL	0-1 FOOT	B	J Q1	Z	228	10716		*1*	9/23/85	
437 -	33- 18-	1-	2	SOIL	LOC. 7 - I-1- 25	CORE VERTICAL	1-2 FEET	B	J,M Q1	Z	229	10717		*1*	9/23/85	
438 -	33- 19-	1-	2	SOIL	LOC. 7 - I-1- 25	CORE VERTICAL	2-3 FEET	B	J,M Q1	Z	230	10718		*1*	9/23/85	
439 -	33- 20-	1-	2	SOIL	LOC. 8 - I-1- 25	CORE VERTICAL	0-1 FOOT	B	J Q1	Z	231	10719		*1*	9/23/85	
440 -	33- 21-	1-	2	SOIL	LOC. 8 - I-1- 25	CORE VERTICAL	1-2 FEET	B	J,M Q1	Z	232	10720		*1*	9/23/85	

441 -	33- 22-	1- 2	SOIL LOC.	8 - I-1- 25	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	233	10721		*1*	9/23/85	
442 -	33- 23-	1- 2	SOIL LOC.	9 - I-1- 25	CORE SURFACE	0-1 FOOT	B	J	Q1	Z	234	10722	DUPL	*1*	9/23/85	19230
443 -	33- 24-	1- 2	SOIL LOC.	10 - I-1- 25	CORE SURFACE	0-1 FOOT	B	J	Q1	Z	235	10723	FWS	*1*	9/23/85	
444 -	33- 25-	1- 2	SOIL LOC.	11 - I-1- 25	CORE SURFACE	0-1 FOOT	B	J	Q1	Z	236	10724		*1*	9/23/85	
445 -	33- 26-	1- 2	SOIL LOC.	12 - I-1- 25	CORE SURFACE	0-1 FOOT	B	J	Q1	Z	237	10725	SPKE	*1*	9/23/85	19270
446 -	33- 27-	1- 4	SOIL LOC.	13 - I-1- 25	CORE SURFACE	0-1 FOOT	D	J	Q1	Z	238	10726		*1*	9/23/85	
447 -	33- 28-	1- 2	SOIL LOC.	14 - I-1- 23	DITCH	0-1 FOOT	B	K	Q1	Z	239	10727		*1*	9/23/85	
448 -	33- 29-	1- 2	SOIL LOC.	15 - I-1- 23	CORE SURFACE	0-6 INCH	B	J	Q1	Z	240	10728	DUPL	*1*	9/23/85	19231
449 -	33- 30-	1- 2	SOIL LOC.	16 - I-1- 64	DITCH	0-1 FOOT	B	K	Q1	Z	241	10729		*1*	9/23/85	
450 -	33- 31-	1- 2	SOIL LOC.	17 - I-1- 64	CORE SURFACE	0-1 FOOT	B	J	Q1	Z	242	10730		*1*	9/23/85	
451 -	33- 32-	1- 2	SOIL LOC.	18 - I-1- 22	DITCH	0-1 FOOT	B	K	Q1	Z	243	10731		*1*	9/23/85	
452 -	33- 33-	1- 2	SOIL LOC.	19 - I-1- 21	DITCH	0-1 FOOT	B	K	Q1	Z	244	10732		*1*	9/23/85	
453 -	33- 34-	1- 2	SOIL LOC.	20 - I-1- 21	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	245	10733	FWS	*1*	9/24/85	
454 -	33- 35-	1- 2	SOIL LOC.	20 - I-1- 21	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	246	10734		*1*	9/24/85	
455 -	33- 36-	1- 2	SOIL LOC.	20 - I-1- 21	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	247	10735		*1*	9/24/85	
456 -	33- 37-	1- 2	SOIL LOC.	21 - I-1- 21	CORE SURFACE	0-1 FOOT	B	J	Q1	Z	248	10736	DUPL	*1*	9/24/85	19232
457 -	33- 38-	1- 2	SOIL LOC.	22 - I-1- 21	CORE SURFACE	0-1 FOOT	B	J	Q1	Z	249	10737		*1*	9/24/85	
458 -	33- 39-	1- 2	SOIL LOC.	23 - STAGING	DITCH	0-1 FOOT	B	K	Q1	Z	250	10738		*1*	9/23/85	
459 -	33- 40-	1- 2	SOIL LOC.	24 - STAGING	DITCH	0-1 FOOT	B	K	Q1	Z	251	10739		*1*	9/23/85	
460 -	33- 41-	1- 2	SOIL LOC.	25 - I-1- 24	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	252	10740		*1*	9/23/85	
461 -	33- 42-	1- 2	SOIL LOC.	25 - I-1- 24	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	253	10741		*1*	9/23/85	
462 -	33- 43-	1- 2	SOIL LOC.	25 - I-1- 24	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	254	10742		*1*	9/23/85	
463 -	33- 44-	1- 2	SOIL LOC.	26 - I-1- 24	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	255	10743		*1*	9/23/85	
464 -	33- 45-	1- 2	SOIL LOC.	26 - I-1- 24	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	256	10744		*1*	9/23/85	
465 -	33- 46-	1- 2	SOIL LOC.	26 - I-1- 24	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	257	10745	SPKE	*1*	9/23/85	19271
466 -	33- 47-	1- 2	SOIL LOC.	27 - I-1- 24	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	258	10746		*1*	9/23/85	
467 -	33- 48-	1- 2	SOIL LOC.	27 - I-1- 24	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	259	10747		*1*	9/24/85	
468 -	33- 49-	1- 2	SOIL LOC.	27 - I-1- 24	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	260	10748		*1*	9/24/85	
469 -	33- 50-	1- 2	SOIL LOC.	28 - I-1- 24	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	261	10749		*1*	9/24/85	
470 -	33- 51-	1- 2	SOIL LOC.	28 - I-1- 24	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	262	10750		*1*	9/24/85	
471 -	33- 52-	1- 2	SOIL LOC.	28 - I-1- 24	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	263	10751		*1*	9/24/85	
472 -	33- 53-	1- 2	SOIL LOC.	29 - I-1- 24	DITCH	0-1 FOOT	B	K	Q1	Z	264	10752		*1*	9/24/85	
473 -	33- 54-	1- 2	SOIL LOC.	30 - I-1- 24	DITCH	0-1 FOOT	B	K	Q1	Z	265	10753	FWS	*1*	9/24/85	
474 -	33- 55-	1- 2	SOIL LOC.	31 - I-1- 24	DITCH	0-1 FOOT	B	K	Q1	Z	266	11641	SPKE	*1*	9/24/85	19272
475 -	33- 56-	1- 2	SOIL LOC.	32 - I-1- 24	DITCH	0-1 FOOT	B	K	Q1	Z	267	11642	DUPL	*1*	9/24/85	19233
476 -	33- 57-	1- 2	SOIL LOC.	33 - I-1- 20	CORE SURFACE	0-1 FOOT	B	J	Q1	Z	268	11643		*1*	9/24/85	
477 -	33- 58-	1- 2	SOIL LOC.	34 - I-1- 20	CORE SURFACE	0-1 FOOT	B	J	Q1	Z	269	11644		*1*	9/24/85	
478 -	33- 59-	1- 2	SOIL LOC.	35 - I-1- 20	CORE SURFACE	0-1 FOOT	B	J	Q1	Z	270	11645		*1*	9/24/85	
479 -	33- 60-	1- 4	SOIL LOC.	36 - I-1- 20	CORE SURFACE	0-1 FOOT	D	J	Q1	Z	271	11646		*1*	9/24/85	
480 -	33- 61-	1- 2	SOIL LOC.	37 - I-1- 19	CORE SURFACE	0-6 INCH	B	J	Q1	Z	272	11647		*1*	9/24/85	

481	-	33-62-	1-2	SOIL LOC.	38 - I-1-	2 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	273	11648		*1*	9/24/85	
482	-	33-63-	1-2	SOIL LOC.	38 - I-1-	2 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	274	11649		*1*	9/24/85	
483	-	33-64-	1-2	SOIL LOC.	38 - I-1-	2 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	275	11650		*1*	9/24/85	
484	-	33-65-	1-2	SOIL LOC.	39 - I-1-	2 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	276	11651	DUPL	*1*	9/24/85	19234
485	-	33-66-	1-2	SOIL LOC.	40 - I-1-	2 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	277	11652	FWS	*1*	9/24/85	
486	-	33-67-	1-2	SOIL LOC.	41 - I-1-	2 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	278	11653		*1*	9/24/85	
487	-	33-68-	1-2	SOIL LOC.	41 - I-1-	2 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	279	11654		*1*	9/24/85	
488	-	33-69-	1-2	SOIL LOC.	41 - I-1-	2 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	280	11655		*1*	9/24/85	
489	-	33-70-	1-2	SOIL LOC.	42 - I-1-	2 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	281	11656		*1*	9/24/85	
490	-	33-71-	1-2	SOIL LOC.	43 - I-1-	2 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	282	11657		*1*	9/24/85	
491	-	33-72-	1-2	SOIL LOC.	44 - I-1-	2 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	283	11658		*1*	9/24/85	
492	-	33-73-	1-2	SOIL LOC.	44 - I-1-	2 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	284	11659		*1*	9/24/85	
493	-	33-74-	1-2	SOIL LOC.	44 - I-1-	2 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	285	11660		*1*	9/24/85	
494	-	33-75-	1-2	SOIL LOC.	45 - I-1-	2 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	286	11661	DUPL	*1*	9/24/85	19235
495	-	33-76-	1-2	SOIL LOC.	56 - I-1-	2 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	287	11662		*1*	9/24/85	
496	-	33-77-	1-2	SOIL LOC.	56 - I-1-	2 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	288	11663		*1*	9/24/85	
497	-	33-78-	1-2	SOIL LOC.	56 - I-1-	2 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	289	11664		*1*	9/24/85	
498	-	33-79-	1-2	SOIL LOC.	57 - I-1-	2 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	290	11665	SPKE	*1*	9/24/85	19273
499	-	33-80-	1-2	SOIL LOC.	58 - I-1-	2 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	291	11666		*1*	9/24/85	
500	-	33-81-	1-2	SOIL LOC.	46 - I-1-	5 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	292	11667		*1*	9/24/85	
501	-	33-82-	1-2	SOIL LOC.	46 - I-1-	5 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	293	11668		*1*	9/24/85	
502	-	33-83-	1-2	SOIL LOC.	46 - I-1-	5 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	294	11669		*1*	9/24/85	
503	-	33-84-	1-2	SOIL LOC.	47 - I-1-	5 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	295	11670		*1*	9/24/85	
504	-	33-85-	1-2	SOIL LOC.	48 - I-1-	5 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	296	11671		*1*	9/24/85	
505	-	33-86-	1-2	SOIL LOC.	49 - I-1-	5 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	297	11672		*1*	9/24/85	
506	-	33-87-	1-2	SOIL LOC.	49 - I-1-	5 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	298	11673		*1*	9/24/85	
507	-	33-88-	1-2	SOIL LOC.	49 - I-1-	5 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	299	11674		*1*	9/24/85	
508	-	33-89-	1-2	SOIL LOC.	50 - I-1-	5 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	300	11675	FWS	*1*	9/24/85	
509	-	33-90-	1-2	SOIL LOC.	51 - I-1-	5 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	301	11676		*1*	9/24/85	
510	-	33-91-	1-2	SOIL LOC.	51 - I-1-	5 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	302	11677		*1*	9/24/85	
511	-	33-92-	1-2	SOIL LOC.	51 - I-1-	5 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	303	11678		*1*	9/24/85	
512	-	33-93-	1-2	SOIL LOC.	52 - I-1-	5 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	304	11679	DUPL	*1*	9/24/85	19236
513	-	33-94-	1-2	SOIL LOC.	53 - I-1-	5 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	305	11680	SPKE	*1*	9/24/85	19274
514	-	33-95-	1-2	SOIL LOC.	54 - I-1-	35 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	306	11681		*1*	9/24/85	
515	-	33-96-	1-2	SOIL LOC.	54 - I-1-	35 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	307	11682		*1*	9/24/85	
516	-	33-97-	1-2	SOIL LOC.	54 - I-1-	35 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	308	11683		*1*	9/24/85	
517	-	33-98-	1-2	SOIL LOC.	55 - I-1-	35 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	309	11684		*1*	9/24/85	
518	-	33-99-	1-2	SOIL LOC.	59 - I-1-	1 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	310	11685	DUPL	*1*	9/24/85	19237
519	-	33-100-	1-2	SOIL LOC.	60 - I-1-	1 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	311	11686		*1*	9/25/85	
520	-	33-101-	1-2	SOIL LOC.	61 - I-1-	1 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	312	11687		*1*	9/25/85	

521	-	33-102-	1- 2	SOIL LOC.	62 - I-1-	3 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	313	11688	SPKE	*1*	9/25/85	19275
522	-	33-103-	1- 2	SOIL LOC.	62 - I-1-	3 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	314	11689		*1*	9/25/85	
523	-	33-104-	1- 2	SOIL LOC.	62 - I-1-	3 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	315	11690		*1*	9/25/85	
524	-	33-105-	1- 2	SOIL LOC.	63 - I-1-	3 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	316	11691	DUPL	*1*	9/25/85	19238
525	-	33-106-	1- 2	SOIL LOC.	63 - I-1-	3 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	317	11692		*1*	9/25/85	
526	-	33-107-	1- 2	SOIL LOC.	63 - I-1-	3 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	318	11693		*1*	9/25/85	
527	-	33-108-	1- 2	SOIL LOC.	64 - I-1-	3 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	319	11694	FWS	*1*	9/24/85	
528	-	33-109-	1- 2	SOIL LOC.	64 - I-1-	3 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	320	11695		*1*	9/24/85	
529	-	33-110-	1- 2	SOIL LOC.	64 - I-1-	3 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	321	11696		*1*	9/24/85	
530	-	33-111-	1- 2	SOIL LOC.	65 - I-1-	3 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	322	11697		*1*	9/24/85	
531	-	33-112-	1- 2	SOIL LOC.	65 - I-1-	3 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	323	11698		*1*	9/24/85	
532	-	33-113-	1- 2	SOIL LOC.	65 - I-1-	3 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	324	11699		*1*	9/24/85	
533	-	33-114-	1- 2	SOIL LOC.	66 - I-1-	3 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	325	11700		*1*	9/24/85	
534	-	33-115-	1- 2	SOIL LOC.	66 - I-1-	3 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	326	11701		*1*	9/24/85	
535	-	33-116-	1- 2	SOIL LOC.	66 - I-1-	3 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	327	11702		*1*	9/24/85	
536	-	33-117-	1- 2	SOIL LOC.	67 - I-1-	3 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	328	11703	DUPL	*1*	9/24/85	19239
537	-	33-118-	1- 2	SOIL LOC.	67 - I-1-	3 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	329	11704		*1*	9/24/85	
538	-	33-119-	1- 2	SOIL LOC.	67 - I-1-	3 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	330	11705		*1*	9/24/85	
539	-	33-120-	1- 2	SOIL LOC.	68 - I-1-	3 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	331	11706	SPKE	*1*	9/24/85	19276
540	-	33-121-	1- 2	SOIL LOC.	68 - I-1-	3 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	332	11707		*1*	9/24/85	
541	-	33-122-	1- 2	SOIL LOC.	68 - I-1-	3 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	333	11708		*1*	9/24/85	
542	-	33-123-	1- 2	SOIL LOC.	69 - I-1-	3 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	334	11709	FWS	*1*	9/24/85	
543	-	33-124-	1- 2	SOIL LOC.	69 - I-1-	3 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	335	11710		*1*	9/24/85	
544	-	33-125-	1- 2	SOIL LOC.	69 - I-1-	3 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	336	11711		*1*	9/24/85	
545	-	33-126-	1- 2	SOIL LOC.	70 - I-1-	3 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	337	11712		*1*	9/25/85	
546	-	33-127-	1- 2	SOIL LOC.	71 - I-1-	3 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	338	11713	DUPL	*1*	9/25/85	19240
547	-	33-128-	1- 2	SOIL LOC.	72 - I-1-	3 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	339	11714		*1*	9/25/85	
548	-	33-129-	1- 4	SOIL LOC.	73 - I-1-	3 CORE SURFACE	0-1 FOOT	D	J	Q1	Z	340	11715		*1*	9/24/85	
549	-	33-130-	1- 2	SOIL LOC.	74 - I-1-	3 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	341	11716	DUPL	*1*	9/25/85	19241
550	-	33-131-	1- 2	SOIL LOC.	75 - I-1-	3 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	342	11717		*1*	9/23/85	
551	-	33-132-	1- 2	SOIL LOC.	75 - I-1-	3 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	343	11718		*1*	9/23/85	
552	-	33-133-	1- 2	SOIL LOC.	75 - I-1-	3 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	344	11719		*1*	9/23/85	
553	-	33-134-	1- 2	SOIL LOC.	76 - I-1-	3 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	345	11720	DUPL	*1*	9/23/85	19242
554	-	33-135-	1- 2	SOIL LOC.	77 - I-1-	3 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	346	11721		*1*	9/23/85	
555	-	33-136-	1- 2	SOIL LOC.	77 - I-1-	3 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	347	11722		*1*	9/23/85	
556	-	33-137-	1- 2	SOIL LOC.	77 - I-1-	3 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	348	11723		*1*	9/23/85	
557	-	33-138-	1- 2	SOIL LOC.	78 - I-1-	3 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	349	11724	SPKE	*1*	9/23/85	19277
558	-	33-139-	1- 2	SOIL LOC.	79 - I-1-	3 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	350	11725	FWS	*1*	9/23/85	
559	-	33-140-	1- 2	SOIL LOC.	80 - I-1-	9 CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	351	11726		*1*	9/24/85	
560	-	33-141-	1- 2	SOIL LOC.	80 - I-1-	9 CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	352	11727		*1*	9/24/85	

561 -	33-142-	1- 2	SOIL LOC.	80 - I-1-	9 CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	353	11728		*1*	9/24/85	
562 -	33-143-	1- 2	SOIL LOC.	81 - I-1-	9 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	354	11729		*1*	9/23/85	
563 -	33-144-	1- 2	SOIL LOC.	82 - I-1-	9 CORE SURFACE	0-1 FOOT	B	J	Q1	Z	355	11730		*1*	9/23/85	
564 -	33-145-	1- 2	SOIL LOC.	83 - I-1-23C	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	356	11731	DUPL	*1*	9/25/85	19243
565 -	33-146-	1- 2	SOIL LOC.	83 - I-1-23C	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	357	11732		*1*	9/25/85	
566 -	33-147-	1- 2	SOIL LOC.	83 - I-1-23C	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	358	11733		*1*	9/25/85	
567 -	33-148-	1- 2	SOIL LOC.	84 - I-1-23C	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	359	11734		*1*	9/25/85	
568 -	33-149-	1- 2	SOIL LOC.	84 - I-1-23C	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	360	11735		*1*	9/25/85	
569 -	33-150-	1- 2	SOIL LOC.	84 - I-1-23C	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	361	11736		*1*	9/25/85	
570 -	33-151-	1- 2	SOIL LOC.	85 - I-1-23C	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	362	11737		*1*	9/25/85	
571 -	33-152-	1- 2	SOIL LOC.	85 - I-1-23C	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	363	11738		*1*	9/25/85	
572 -	33-153-	1- 2	SOIL LOC.	85 - I-1-23C	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	364	11739		*1*	9/25/85	
573 -	33-154-	1- 2	SOIL LOC.	86 - I-1-23C	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	365	11740		*1*	9/25/85	
574 -	33-155-	1- 2	SOIL LOC.	86 - I-1-23C	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	366	11741	DUPL	*1*	9/25/85	19244
575 -	33-156-	1- 2	SOIL LOC.	86 - I-1-23C	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	367	11742		*1*	9/25/85	
576 -	33-157-	1- 4	SOIL LOC.	87 - I-1-23C	CORE VERTICAL	0-1 FOOT	D	J	Q1	Z	368	11743		*1*	9/25/85	
577 -	33-158-	1- 2	SOIL LOC.	87 - I-1-23C	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	369	11744		*1*	9/25/85	
578 -	33-159-	1- 2	SOIL LOC.	87 - I-1-23C	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	370	11745		*1*	9/25/85	
579 -	33-160-	1- 2	SOIL LOC.	88 - I-1-23C	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	371	11746		*1*	9/25/85	
580 -	33-161-	1- 2	SOIL LOC.	88 - I-1-23C	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	372	11747		*1*	9/25/85	
581 -	33-162-	1- 2	SOIL LOC.	88 - I-1-23C	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	373	11748		*1*	9/25/85	
582 -	33-163-	1- 2	SOIL LOC.	89 - I-1-23C	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	374	11749	DUPL	*1*	9/25/85	19245
583 -	33-164-	1- 2	SOIL LOC.	89 - I-1-23C	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	375	11750		*1*	9/25/85	
584 -	33-165-	1- 2	SOIL LOC.	89 - I-1-23C	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	376	11751		*1*	9/25/85	
585 -	33-166-	1- 2	SOIL LOC.	90 - I-1-23C	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	377	11752	FWS	*1*	9/25/85	
586 -	33-167-	1- 2	SOIL LOC.	90 - I-1-23C	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	378	11753		*1*	9/25/85	
587 -	33-168-	1- 2	SOIL LOC.	90 - I-1-23C	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	379	11754	SPKE	*1*	9/25/85	19278
588 -	33-169-	1- 2	SOIL LOC.	91 - I-1-23C	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	380	11755		*1*	9/25/85	
589 -	33-170-	1- 2	SOIL LOC.	91 - I-1-23C	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	381	11756	SPKE	*1*	9/25/85	19279
590 -	33-171-	1- 2	SOIL LOC.	91 - I-1-23C	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	382	11757		*1*	9/25/85	
591 -	33-172-	1- 2	SOIL LOC.	92 - I-1-23C	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	383	11758		*1*	9/25/85	
592 -	33-173-	1- 2	SOIL LOC.	92 - I-1-23C	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	384	11759		*1*	9/25/85	
593 -	33-174-	1- 2	SOIL LOC.	92 - I-1-23C	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	385	11760	DUPL	*1*	9/25/85	19246
594 -	33-175-	1- 2	SOIL LOC.	93 - I-1-23C	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	386	11761		*1*	9/25/85	
595 -	33-176-	1- 2	SOIL LOC.	93 - I-1-23C	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	387	11762		*1*	9/25/85	
596 -	33-177-	1- 2	SOIL LOC.	93 - I-1-23C	CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	388	11763		*1*	9/25/85	
597 -	33-178-	1- 2	SOIL LOC.	94 - I-1-23C	CORE SURFACE	0-1 FOOT	B	J	Q1	Z	389	11764		*1*	9/25/85	
598 -	33-179-	1- 2	SOIL LOC.	95 - I-1-23C	CORE SURFACE	0-1 FOOT	B	J	Q1	Z	390	11765		*1*	9/25/85	
599 -	33-180-	1- 2	SOIL LOC.	96 - I-1-23C	CORE VERTICAL	0-1 FOOT	B	J	Q1	Z	391	11766	DUPL	*1*	9/25/85	19247
600 -	33-181-	1- 2	SOIL LOC.	96 - I-1-23C	CORE VERTICAL	1-2 FEET	B	J,M	Q1	Z	392	11767		*1*	9/25/85	

601 -	33-182-	1-	2	SOIL LOC. 96 - I-1-23C CORE VERTICAL	2-3 FEET	B	J,M	Q1	Z	393	11768		*1*	9/25/85
602 -	33-183-	1-	2	SOIL LOC. 97 - I-1- 9 SURFACE COMP.	0-1 FOOT	B	J	Q1	X	394	11769		*1*	9/25/85
603 -	33-184-	1-	2	SOIL LOC. 98 - I-1- 15 SURFACE COMP.	0-1 FOOT	B	J	Q1	X	395	11770		*1*	9/25/85
604 -	33-185-	1-	2	SOIL LOC. 99 - I-1- 1 SURFACE COMP.	0-1 FOOT	B	J	Q1	X	396	11771		*1*	9/25/85
605 -	33-186-	1-	2	SOIL LOC. 100 - I-1- 29 SURFACE COMP.	0-1 FOOT	B	J	Q1	X	397	11772		*1*	9/25/85
606 -	33-187-	1-	2	SOIL LOC. 101 - I-1- 8 SURFACE COMP.	0-1 FOOT	B	J	Q1	X	398	11773	DUPL	*1*	9/25/85 19248
607 -	33-188-	1-	2	SOIL LOC. 102 - I-1- 8 SURFACE COMP.	0-1 FOOT	B	J	Q1	X	399	11774	FWS	*1*	9/25/85

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 GROUP: #11 SITE: 35:AREA 9 EAST WATERWAY  
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614 -	35-	1-	3-	1 SEDIMENT	WATERWAY	COMP. 5 GRABS	0-1 FT	A	I	P	Y	548	19208	*1*	8/13/85
615 -	35-	1-	3-	6 SEDIMENT	WATERWAY	COMP. 5 GRABS	0-1 FT	F	I	P	Y	598	9283	*1*	11/18/85

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 GROUP: #12 SITE: 34:CRAB ORCHARD LAKE  
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622 -	34-	1-	2-	5	WATER REFUGE INTAKE	GRAB	NA	E	-	T	W	413	3252	*1*	7/24/85
623 -	34-	2-	2-	5	WATER MARION INTAKE	GRAB	NA	E	-	T	W	414	3251	*1*	7/24/85
624 -	34-	3-	2-	5	WATER MARION RES.-INTAKE	GRAB	NA	E	-	T	W	415	3253	*1*	7/25/85 Resamp. 9/24/85
625 -	34-	4-	2-	5	WATER REFUGE TREATED H2O	GRAB	NA	E	-	T	W	416	3246	*1*	7/24/85 Resamp. 9/24/85
626 -	34-	5-	2-	5	WATER MARION TREATED H2O	GRAB	NA	E	-	T	W	417	3255	*1*	7/25/85 Resamp. 9/24/85
627 -	34-	6-	2-	9	WATER LAKE 1 B	COMP.3 DEPTHS	SURF-0.8 FT	I	O	S	W	418	11788	.1.	
628 -	34-	7-	2-	9	WATER LAKE 2 C	COMP.3 DEPTHS	SURF-0.8 FT	I	O	S	W	419	11789	DUPL	.1. 19218
629 -	34-	8-	2-	9	WATER LAKE 3 G	COMP.3 DEPTHS	SURF-0.8 FT	I	O	S	W	420	11790	.1.	
630 -	34-	9-	2-	9	WATER LAKE 4 H	COMP.3 DEPTHS	SURF-0.8 FT	I	O	S	W	421	11791	.1.	
631 -	34-	10-	2-	9	WATER LAKE 5 A	COMP.3 DEPTHS	SURF-0.8 FT	I	O	S	W	422	11792	.1.	
632 -	34-	11-	2-	9	WATER LAKE 6 D	COMP.3 DEPTHS	SURF-0.8 FT	I	O	S	W	423	11793	SPKE	.1. 19265
633 -	34-	12-	2-	9	WATER LAKE 7 E	COMP.3 DEPTHS	SURF-0.8 FT	I	O	S	W	424	11794	.1.	
634 -	34-	13-	2-	9	WATER LAKE 8 F	COMP.3 DEPTHS	SURF-0.8 FT	I	O	S	W	425	11795	.1.	
635 -	34-	14-	2-	9	WATER LAKE 9 I	COMP.3 DEPTHS	SURF-0.8 FT	I	O	S	W	426	11796	.1.	
636 -	34-	15-	2-	9	WATER LAKE 10 J	COMP.3 DEPTHS	SURF-0.8 FT	I	O	S	W	427	19159	.1.	
637 -	34-	16-	3-	8	SEDIMENT LAKE 1 B	GRAB	DREDGE	H	K	S	W	428	19160	.1.	
638 -	34-	17-	3-	8	SEDIMENT LAKE 2 C	GRAB	DREDGE	H	K	S	W	429	19161	DUPL	.1. 9286
639 -	34-	18-	3-	9	SEDIMENT LAKE 3 G	GRAB	DREDGE	I	K	S	W	430	19162	FWS	.1.
640 -	34-	19-	3-	9	SEDIMENT LAKE 4 H	GRAB	DREDGE	I	K	S	W	431	19163	.1.	

641 -	34- 20-	3- 9	SEDIMENT	LAKE 5 A	GRAB	DREDGE	I	K	S	W	432	19164	SPKE	.1.	19286
642 -	34- 21-	3- 9	SEDIMENT	LAKE 6 D	GRAB	DREDGE	I	K	S	W	433	19165	DUPL	.1.	19260
643 -	34- 22-	3- 9	SEDIMENT	LAKE 7 E	GRAB	DREDGE	I	K	S	W	434	19166	DUPL	.1.	19261
644 -	34- 23-	3- 9	SEDIMENT	LAKE 8 F	GRAB	DREDGE	I	K	S	W	435	19167		.1.	
645 -	34- 24-	3- 9	SEDIMENT	LAKE 9 I	GRAB	DREDGE	I	K	S	W	436	19168		.1.	
646 -	34- 25-	3- 9	SEDIMENT	LAKE 10J	GRAB	DREDGE	I				437	19169		.1.	
647 -	34- 26-	4- 20	FISH	LAKE SITE 1 B	COMP. 5 CARP	NA	T				438	19170		#1.	7/23/85
648 -	34- 27-	4- 20	FISH	LAKE SITE 1 B	COMP. 5 BASS	NA	T				439	19171		#1.	7/23/85
649 -	34- 28-	4- 20	FISH	LAKE SITE 1 B	COMP. 5 BASS	NA	T				440	19172		#1.	7/23/85
650 -	34- 48-	4- 20	FISH	LAKE SITE 1 B	COMP.5 BULLHEAD	NA	T				460	19192	FWS	#1.	7/23/85
651 -	34- 49-	4- 20	FISH	LAKE SITE 1 B	COMP.5 BULLHEAD	NA	T				461	19193		#1.	7/23/85
652 -	34- 50-	4- 20	FISH	LAKE SITE 1 B	COMP.2 CATFISH	NA	T				462	19194		#1.	7/23/85
653 -	34- 29-	4- 8	FISH	LAKE SITE 2 C	COMP. 5 CARP	NA	H				441	19173	FWS	#1.	7/23/85
654 -	34- 30-	4- 20	FISH	LAKE SITE 2 C	COMP. 5 CARP	NA	T				442	19174		#1.	7/23/85
655 -	34- 31-	4- 8	FISH	LAKE SITE 2 C	COMP. 5 BASS	NA	H				443	19175		#1.	7/23/85
656 -	34- 51-	4- 20	FISH	LAKE SITE 2 C	COMP.5 BULLHEAD	NA	T				463	19195		#1.	7/23/85
657 -	34- 52-	4- 20	FISH	LAKE SITE 2 C	COMP.5 BULLHEAD	NA	T				464	19196		#1.	7/23/85
658 -	34- 53-	4- 20	FISH	LAKE SITE 2 C	COMP.5 CATFISH	NA	T				465	19197		#1.	7/23/85
659 -	34- 32-	4- 20	FISH	LAKE SITE 3 G	COMP. 5 CARP	NA	T				444	19176		#1.	7/23/85
660 -	34- 33-	4- 20	FISH	LAKE SITE 3 G	COMP. 5 CARP	NA	T				445	19177		#1.	7/23/85
661 -	34- 34-	4- 20	FISH	LAKE SITE 3 G	COMP. 5 BASS	NA	T				446	19178	FWS	#1.	7/23/85
662 -	34- 54-	4- 20	FISH	LAKE SITE 3 G	COMP.5 BULLHEAD	NA	T				466	19198		#1.	7/23/85
663 -	34- 55-	4- 20	FISH	LAKE SITE 3 G	COMP.5 BULLHEAD	NA	T				467	19199		#1.	7/23/85
664 -	34- 35-	4- 20	FISH	LAKE SITE 4 H	COMP. 5 CARP	NA	T				447	19179	FWS	#1.	7/24/85
665 -	34- 36-	4- 20	FISH	LAKE SITE 4 H	COMP. 5 BASS	NA	T				448	19180		#1.	7/24/85
666 -	34- 37-	4- 20	FISH	LAKE SITE 4 H	COMP. 5 BASS	NA	T				449	19181		#1.	7/24/85
667 -	34- 59-	4- 20	FISH	LAKE SITE 4 H	COMP.5 BULLHEAD	NA	T				472	19200		#1.	7/24/85
668 -	34- 60-	4- 20	FISH	LAKE SITE 4 H	COMP.5 BULLHEAD	NA	T				473	19201		#1.	7/24/85
669 -	34- 61-	4- 20	FISH	LAKE SITE 4 H	COMP.4 CATFISH	NA	T				471	19202		#1.	7/24/85
670 -	34- 38-	4- 20	FISH	LAKE CONTROL J	COMP. 5 CARP	NA	T				450	19182		#1.	7/24/85
671 -	34- 39-	4- 20	FISH	LAKE CONTROL J	COMP. 5 CARP	NA	T				451	19183		#1.	7/24/85
672 -	34- 40-	4- 20	FISH	LAKE CONTROL J	COMP. 3 BASS	NA	T				452	19184	FWS	#1.	7/24/85
673 -	34- 41-	4- 20	FISH	LAKE CONTROL J	COMP. 5 BASS	NA	T				453	19185		#1.	7/24/85
674 -	34- 56-	4- 20	FISH	LAKE CONTROL J	COMP.5 BULLHEAD	NA	T				468	19203		#1.	7/24/85
675 -	34- 57-	4- 20	FISH	LAKE CONTROL J	COMP.5 BULLHEAD	NA	T				469	19204		#1.	7/24/85
676 -	34- 58-	4- 20	FISH	LAKE CONTROL J	COMP.3 CATFISH	NA	T				470	19205		#1.	7/24/85
677 -															
678 -															
679 -															
680 -															

GROUP: #13 SITE: 31:REFUGE CONTROL SITE

681 -	-----																
682 -																	
683 -	31-	1-	1-	4	SOIL	REFUGE CONTROL	SINGLE SAMPLE	SURFACE	D	--	T	W	474	19206	*1*	8/14/85	Near dead tree
684 -	31-	1-	1-	7	SOIL	REFUGE CONTROL	SINGLE SAMPLE	SURFACE	G	--	T	W	599	9284	*1*	11/19/85	
685 -	31-	2-	2-	9	WATER	REFUGE CONTROL	SINGLE SAMPLE	BAILER	I	--	T	W	475	19207	.1.		
686 -																	
687 -																	
688 -	-----																
689 -	GROUP: #14 SITE: 40:DUPLICATES																
690 -	-----																
691 -																	
692 -	40-	1-	2-	1	WATER	DUPLICATE			A				476	19215 DUPL	*1*	8/13/85	9467
693 -	40-	3-	2-	9	WATER	DUPLICATE			I				478	19217 DUPL	.1.		10697
694 -	40-	4-	2-	9	WATER	DUPLICATE			I				479	19218 DUPL	.1.		11789
695 -	40-	5-	2-	17	WATER	DUPLICATE-WELL			Q				480	19219 DUPL	.1.		9450
696 -	40-	6-	2-	19	WATER	DUPLICATE-WELL			S				481	19220 DUPL	.1.		9494
697 -	40-	7-	1-	1	SOIL	DUPLICATE			A				482	19221 DUPL	*1*	8/14/85	9402
698 -	40-	8-	1-	6	SOIL	DUPLICATE			F				483	19222 DUPL	*1*	11/19/85	9251
699 -	40-	9-	1-	1	SOIL	DUPLICATE			A				484	19223 DUPL	*1*	8/15/85	9453
700 -	40-	10-	1-	1	SOIL	DUPLICATE			A				485	19224 DUPL	*1*	8/14/85	9463
701 -	40-	11-	3-	6	SEDIMENT	DUPLICATE			F				486	19225 DUPL	*1*	11/18/85	9261
702 -	40-	12-	1-	1	SOIL	DUPLICATE			A				487	19226 DUPL	*1*	8/17/85	9417
703 -	40-	13-	1-	7	SOIL	DUPLICATE			G				488	19227 DUPL	*1*	11/18/85	9270
704 -	40-	14-	1-	2	SOIL	DUPLICATE			B				489	19228 DUPL	*1*	9/23/85	10703
705 -	40-	15-	1-	2	SOIL	DUPLICATE			B				490	19229 DUPL	*1*	9/23/85	10715
706 -	40-	16-	1-	2	SOIL	DUPLICATE			B				491	19230 DUPL	*1*	9/23/85	10722
707 -	40-	17-	1-	2	SOIL	DUPLICATE			B				492	19231 DUPL	*1*	9/23/85	10728
708 -	40-	18-	1-	2	SOIL	DUPLICATE			B				493	19232 DUPL	*1*	9/24/85	10736
709 -	40-	19-	1-	2	SOIL	DUPLICATE			B				494	19233 DUPL	*1*	9/23/85	11642
710 -	40-	20-	1-	2	SOIL	DUPLICATE			B				495	19234 DUPL	*1*	9/24/85	11651
711 -	40-	21-	1-	2	SOIL	DUPLICATE			B				496	19235 DUPL	*1*	9/24/85	11661
712 -	40-	22-	1-	2	SOIL	DUPLICATE			B				497	19236 DUPL	*1*	9/24/85	11679
713 -	40-	23-	1-	2	SOIL	DUPLICATE			B				498	19237 DUPL	*1*	9/24/85	11685
714 -	40-	24-	1-	2	SOIL	DUPLICATE			B				499	19238 DUPL	*1*	9/25/85	11691
715 -	40-	25-	1-	2	SOIL	DUPLICATE			B				500	19239 DUPL	*1*	9/24/85	11703
716 -	40-	26-	1-	2	SOIL	DUPLICATE			B				501	19240 DUPL	*1*	9/25/85	11713
717 -	40-	27-	1-	2	SOIL	DUPLICATE			B				502	19241 DUPL	*1*	9/25/85	11716
718 -	40-	28-	1-	2	SOIL	DUPLICATE			B				503	19242 DUPL	*1*	9/23/85	11719
719 -	40-	29-	1-	2	SOIL	DUPLICATE			B				504	19243 DUPL	*1*	9/25/85	11731
720 -	40-	30-	1-	2	SOIL	DUPLICATE			B				505	19244 DUPL	*1*	9/25/85	11741



721 -	40- 31- 1- 2	SOIL	DUPLICATE	B	506	19245 DUPL	*1*	9/25/85	11749
722 -	40- 32- 1- 2	SOIL	DUPLICATE	B	507	19246 DUPL	*1*	9/25/85	11760
723 -	40- 33- 1- 2	SOIL	DUPLICATE	B	508	19247 DUPL	*1*	9/25/85	11766
724 -	40- 34- 1- 2	SOIL	DUPLICATE	B	509	19248 DUPL	*1*	9/25/85	11773
725 -	40- 35- 1- 3	SOIL	DUPLICATE	C	510	19249 DUPL	*1*	8/22/85	10654
726 -	40- 36- 1- 3	SOIL	DUPLICATE	C	511	19250 DUPL	*1*	8/24/85	9500
727 -	40- 37- 1- 3	SOIL	DUPLICATE	C	512	19251 DUPL	*1*	8/22/85	10660
728 -	40- 38- 1- 8	SOIL	DUPLICATE	H	513	19252 DUPL	*1*	8/22/85	10661
729 -	40- 39- 1- 4	SOIL	DUPLICATE	D	514	19253 DUPL	*1*	8/17/85	9398
730 -	40- 40- 3- 1	SEDIMENT	DUPLICATE	A	515	19254 DUPL	*1*	8/16/85	9429
731 -	40- 41- 3- 1	SEDIMENT	DUPLICATE	A	516	19255 DUPL	*1*	8/16/85	19210
732 -	40- 42- 3- 1	SEDIMENT	DUPLICATE	A	517	19256 DUPL	*1*	8/13/85	9468
733 -	40- 43- 3- 1	SEDIMENT	DUPLICATE	A	518	19257 DUPL	*1*	8/22/85	10685
734 -	40- 44- 3- 1	SEDIMENT	DUPLICATE	A	519	19258 DUPL	*1*	8/23/85	10691
735 -	40- 45- 3- 7	SEDIMENT	DUPLICATE	G	520	19259 DUPL	*1*	11/19/85	9262
736 -	40- 46- 3- 9	SEDIMENT	DUPLICATE	I	521	19260 DUPL	.1.		19165
737 -	40- 47- 3- 9	SEDIMENT	DUPLICATE	I	522	19261 DUPL	.1.		19166
738 -	40- 48- 1- 1	SOIL	DUPLICATE	A	549	19287 DUPL	*1*	8/13/85	9491
739 -	40- 49- 1- 4	SOIL	DUPLICATE	D	551	19289 DUPL	*1*	8/16/85	9461
740 -	40- 50- 1- 4	SOIL	DUPLICATE	D	552	19290 DUPL	*1*	8/16/85	9443
741 -	40- 51- 1- 4	SOIL	DUPLICATE	D	553	19291 DUPL	*1*	8/14/85	9473
742 -	40- 52- 3- 1	SEDIMENT EXPLOSIVES ANAL.	DUPLICATE	A	555	9255 DUPL	*1*	7/25/85	3387
743 -	40- 53- 3- 8	SEDIMENT	DUPLICATE	H	601	9286 DUPL	.1.		19161
744 -									
745 -									
746 -									
747 -									
748 -									
749 -									
750 -	41- 1- 3- 6	SEDIMENT	SPIKE	F	523	19262 SPKE	*1*	12/05/85	9278
751 -	41- 2- 1- 7	SOIL	SPIKE	G	524	19263 SPKE	*1*	11/19/85	9281
752 -	41- 3- 2- 9	WATER	SPIKE	I	525	19264 SPKE	.1.		19161
753 -	41- 4- 2- 19	WATER	SPIKE -WELL	S	526	19265 SPKE	.1.		11793
754 -	41- 5- 1- 1	SOIL	SPIKE	A	527	19266 SPKE	*1*	8/14/85	9403
755 -	41- 6- 1- 1	SOIL	SPIKE	A	528	19267 SPKE	*1*	8/17/85	9436
756 -	41- 7- 1- 1	SOIL	SPIKE	A	529	19268 SPKE	*1*	8/16/85	9459
757 -	41- 8- 1- 1	SOIL	SPIKE	A	530	19269 SPKE	*1*	8/14/85	9481
758 -	41- 9- 1- 2	SOIL	SPIKE	B	531	19270 SPKE	*1*	9/23/85	10725
759 -	41- 10- 1- 2	SOIL	SPIKE	B	532	19271 SPKE	*1*	9/23/85	10745
760 -	41- 11- 1- 2	SOIL	SPIKE	B	533	19272 SPKE	*1*	9/23/85	11641

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 GROUP: #14 SITE: 41:SPIKES  
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761 -	41- 12- 1- 2	SOIL	SPIKE	B	534	19273 SPKE	*1*	9/24/85	11665
762 -	41- 13- 1- 2	SOIL	SPIKE	B	535	19274 SPKE	*1*	9/24/85	11680
763 -	41- 14- 1- 2	SOIL	SPIKE	B	536	19275 SPKE	*1*	9/25/85	11688
764 -	41- 15- 1- 2	SOIL	SPIKE	B	537	19276 SPKE	*1*	9/24/85	11706
765 -	41- 16- 1- 2	SOIL	SPIKE	B	538	19277 SPKE	*1*	9/23/85	11724
766 -	41- 17- 1- 2	SOIL	SPIKE	B	539	19278 SPKE	*1*	9/25/85	11754
767 -	41- 18- 1- 2	SOIL	SPIKE	B	540	19279 SPKE	*1*	9/25/85	11756
768 -	41- 19- 1- 3	SOIL	SPIKE	C	541	19280 SPKE	*1*	8/23/85	10666
769 -	41- 20- 1- 8	SOIL	SPIKE	H	542	19281 SPKE	*1*	8/23/85	10665
770 -	41- 21- 3- 1	SEDIMENT	SPIKE	A	543	19282 SPKE	*1*	8/16/85	9427
771 -	41- 24- 3- 4	SEDIMENT	SPIKE	D	546	19285 SPKE	*1*	8/23/85	10694
772 -	41- 25- 3- 9	SEDIMENT	SPIKE	I	547	19286 SPKE	.1.		19164
773 -	41- 26- 1- 1	SOIL	SPIKE	A	550	19288 SPKE	*1*	8/13/85	9487
774 -	41- 27- 1- 4	SOIL EXPLOSIVES ANAL.	SPIKE	D	557	46700 SPKE	*1*	7/25/85	3385
775 -	41- 28- 1- 4	SOIL	SPIKE	D	561	9256 SPKE	*1*	7/25/85	46700
776 -	41- 29- 1- 1	SOIL	SPIKE	A	562	85576 SPKE	*1*	8/13/85	9467
777 -	41- 30- 1- 1	SOIL	SPIKE	A	569	14138 SPKE	*1*	8/14/85	9463

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 GROUP: #14 SITE: 42:BLANKS  
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784 -	42- 1- 1- 4	SOIL FIELD - SAND	BLANK	D	554	19292 BLNK	*1*	8/19/85	
785 -	42- 2- 1- 1	SOIL EXPLOSIVES ANAL.	BLANK	A	556	46699 BLNK	*1*	8/19/85	
786 -	42- 3- 1- 1	SOIL OR&G LAB	BLANK	A	563	85575 BLNK	*1*	8/19/85	
787 -	42- 4- 1- 1	SOIL OR&G LAB	BLANK	A	564	46453 BLNK	*1*	8/19/85	
788 -	42- 5- 1- 1	SOIL OR&G LAB	BLANK	A	565	85608 BLNK	*1*	8/19/85	
789 -	42- 6- 1- 1	SOIL OR&G LAB	BLANK	A	566	14139 BLNK	*1*	8/19/85	
790 -	42- 7- 1- 1	SOIL OR&G LAB	BLANK	A	567	2994 BLNK	*1*	8/19/85	
791 -	42- 8- 1- 1	SOIL OR&G LAB	BLANK	A	568	2995 BLNK	*1*	8/19/85	
792 -	42- 9- 1- 1	SOIL OR&G LAB	BLANK	A	570	46508 BLNK	*1*	8/23/85	
793 -	42- 10- 1- 1	SOIL OR&G LAB	BLANK	A	571	46683 BLNK	*1*	8/28/85	
794 -	42- 11- 1- 7	SOIL OR&G LAB	BLANK	G	600	9285 BLNK	*1*	11/20/85	
795 -	42- 12- 1- 7	SOIL OR&G LAB	BLANK	G	602	9287 BLNK	.1.		
796 -	42- 13- 1- 8	SOIL OR&G LAB	BLANK	H	603	9288 BLNK	.1.		
797 -	42- 14- 1- 9	SOIL OR&G LAB	BLANK	I	604	9289 BLNK	.1.		

798 -  
 799 -  
 800 -

PHASE II LISTING OF SAMPLES SCHEDULED

## CRAB ORCHARD NATIONAL WILDLIFE REFUGE

## SAMPLING AND ANALYSIS SCHEDULE

Revised March 17, 1986

## PHASE II

ID1	ID2	ID3	ID4	MATRIX!	NAME	TYPE	DEPTH	ANAL!	DEPTH!	LOCA!	INTRVL!	SAMP!	LAB	REPLICATE	SAMPLE	DUPL./SPIKE	NOTES
ST	N	MAT	A.S					SET	TION	& NO.	NOS.	NO	LAB	FWS	COLL.	DATE	NUMBERS
!....(RATIONALE)....!																	
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GROUP: #2 SITE: 10:WATERWORKS NORTH DRAINAGE																	
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10-	3-	2-	10	WATER	WM-N WATER -1	COMP. 4 GRABS	SURFACE	J					605	66601		.2.	
10-	4-	3-	10	SEDIMENT	WM-N SEDIMENT-1	GRAB	0-1 FT	J					606	66602	DUPL	.2.	67440
10-	5-	3-	10	SEDIMENT	WM-N SEDIMENT-2	GRAB	0-1 FT	J					607	66603		.2.	
10-	6-	3-	10	SEDIMENT	WM-N SEDIMENT-3	GRAB	0-1 FT	J					608	66604		.2.	
10-	7-	3-	10	SEDIMENT	WM-N SEDIMENT-4	GRAB	0-1 FT	J					609	66605		.2.	
10-	8-	3-	10	SEDIMENT	WM-N SEDIMENT-5	GRAB	0-1 FT	J					610	66606		.2.	
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GROUP: #2 SITE: 11:P AREA SOUTHEAST DRAINAGE																	
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11-	3-	3-	11	SEDIMENT	P-SE SEDIMENT-1	GRAB	0-1 FT	K					611	66607		.2.	
11-	4-	3-	11	SEDIMENT	P-SE SEDIMENT-2	GRAB	0-1 FT	K					612	66608		.2.	
11-	5-	3-	11	SEDIMENT	P-SE SEDIMENT-3	GRAB	0-1 FT	K					613	66609		.2.	
11-	6-	3-	11	SEDIMENT	P-SE SEDIMENT-4	GRAB	0-1 FT	K					614	66610		.2.	
11-	7-	3-	11	SEDIMENT	P-SE SEDIMENT-5	GRAB	0-1 FT	K					615	66611	DUPL	.2.	67441
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GROUP: #3 SITE: 14:AREA 14 SOLVENT STORAGE																	
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14-	5-	3-	12	SEDIMENT	DITCH -1	GRAB	0-1 FT	L					616	66612		.2.	
14-	6-	3-	12	SEDIMENT	DITCH -2	GRAB	0-1 FT	L					617	66613	DUPL	.2.	67442
14-	7-	3-	12	SEDIMENT	DITCH -3	GRAB	0-1 FT	L					618	66614		.2.	
14-	8-	3-	12	SEDIMENT	DITCH -4	GRAB	0-1 FT	L					619	66615		.2.	
14-	9-	3-	12	SEDIMENT	DITCH -5	GRAB	0-1 FT	L					620	66616		.2.	

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 GROUP: #4 SITE: 15:AREA 7 PLATING POND
 

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15-	4-	3- 14	SEDIMENT	PLATING POND	COMP. 4 GRABS	0-1 FT	N	621	66617	.2.
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 GROUP: #4 SITE: 16:AREA 7 INDUSTRIAL SITE
 

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16-	18-	2- 15	WATER	DITCH NO.3	COMP. 2 GRABS	SURFACE	0	622	66618	.2.
16-	19-	2- 15	WATER	DITCH NO.4	COMP. 2 GRABS	SURFACE	0	623	66619	.2.

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 GROUP: #5 SITE: 17:JOB CORPS LANDFILL
 

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17-	14-	2- 17	WATER	POND NO.1	GRAB	SURFACE	Q	624	66620	.2.	
17-	15-	2- 17	WATER	POND NO.2	GRAB	SURFACE	Q	625	66621	.2.	
17-	16-	3- 17	SEDIMENT	POND NO.1	GRAB	0-1 FT	Q	626	66622	.2.	
17-	17-	3- 17	SEDIMENT	POND NO.2	GRAB	0-1 FT	Q	627	66623	.2.	
17-	18-	1- 16	SOIL	SQUARE 1-SW 1	GRAB	SURFACE	P	628	66624 DUPL	.2.	67443
17-	19-	1- 16	SOIL	SQUARE 1-SW 1	GRAB	3 FT	P	629	66625 FWS	.2.	
17-	20-	1- 16	SOIL	SQUARE 1-SE 2	GRAB	SURFACE	P	630	66626 SPKE	.2.	67444
17-	21-	1- 16	SOIL	SQUARE 1-SE 2	GRAB	3 FT	P	631	66627	.2.	
17-	22-	1- 16	SOIL	SQUARE 1-NE 3	GRAB	SURFACE	P	632	66628 DUPL	.2.	67445
17-	23-	1- 16	SOIL	SQUARE 1-NE 3	GRAB	3 FT	P	633	66629	.2.	
17-	24-	1- 16	SOIL	SQUARE 1-NW 4	GRAB	SURFACE	P	634	66630	.2.	
17-	25-	1- 16	SOIL	SQUARE 1-NW 4	GRAB	3 FT	P	635	66631 FWS	.2.	
17-	26-	1- 17	SOIL	SQUARE 2-S 5	GRAB	SURFACE	Q	636	66632	.2.	
17-	27-	1- 17	SOIL	SQUARE 2-S 5	GRAB	SURFACE	Q	637	66633	.2.	
17-	28-	1- 16	SOIL	SQUARE 2-S 6	GRAB	SURFACE	P	638	66634	.2.	
17-	29-	1- 16	SOIL	SQUARE 2-S 7	GRAB	SURFACE	P	639	66635 FWS	.2.	
17-	30-	1- 17	SOIL	SQUARE 2-S 8	GRAB	SURFACE	Q	640	66636 DUPL	.2.	67446
17-	31-	1- 17	SOIL	SQUARE 2-S 8	GRAB	3 FT	Q	641	66637	.2.	
17-	32-	1- 16	SOIL	SQUARE 2-S 9	GRAB	SURFACE	P	642	66638	.2.	

81 -	17- 33-	1- 16	SOIL	SQUARE 2-E 10	GRAB	SURFACE	P	643	66639	.2.	
82 -	17- 34-	1- 17	SOIL	SQUARE 2-N 11	GRAB	SURFACE	Q	644	66640 DUPL	.2.	67447
83 -	17- 35-	1- 17	SOIL	SQUARE 2-N 11	GRAB	3 FT	Q	645	66641	.2.	
84 -	17- 36-	1- 16	SOIL	SQUARE 2-N 12	GRAB	SURFACE	P	646	66642	.2.	
85 -	17- 37-	1- 16	SOIL	SQUARE 2-N 13	GRAB	SURFACE	P	647	66643	.2.	
86 -	17- 38-	1- 17	SOIL	SQUARE 2-N 14	GRAB	SURFACE	Q	648	66644	.2.	
87 -	17- 39-	1- 17	SOIL	SQUARE 2-N 14	GRAB	3 FT	Q	649	66645	.2.	
88 -	17- 40-	1- 16	SOIL	SQUARE 2-W 15	GRAB	SURFACE	P	650	66646	.2.	
89 -	17- 41-	1- 16	SOIL	SQUARE 2-W 15	GRAB	3 FT	P	651	66647	.2.	
90 -	17- 42-	1- 16	SOIL	SQUARE 2-W 16	GRAB	SURFACE	P	652	66648 DUPL	.2.	67448
91 -	17- 43-	1- 17	SOIL	SQUARE 3-S 17	GRAB	SURFACE	Q	653	66649 FMS	.2.	
92 -	17- 44-	1- 16	SOIL	SQUARE 3-S 18	GRAB	SURFACE	P	654	66650	.2.	
93 -	17- 45-	1- 16	SOIL	SQUARE 3-S 19	GRAB	SURFACE	P	655	66651	.2.	
94 -	17- 46-	1- 16	SOIL	SQUARE 3-S 20	GRAB	SURFACE	P	656	66652	.2.	
95 -	17- 47-	1- 16	SOIL	SQUARE 3-S 21	GRAB	SURFACE	P	657	66653	.2.	
96 -	17- 48-	1- 16	SOIL	SQUARE 3-S 22	GRAB	SURFACE	P	658	66654	.2.	
97 -	17- 49-	1- 16	SOIL	SQUARE 3-E 23	GRAB	SURFACE	P	659	66655	.2.	
98 -	17- 50-	1- 16	SOIL	SQUARE 3-N 24	GRAB	SURFACE	P	660	66656 SPKE	.2.	67449
99 -	17- 51-	1- 16	SOIL	SQUARE 3-N 25	GRAB	SURFACE	P	661	66657	.2.	
100 -	17- 52-	1- 16	SOIL	SQUARE 3-N 26	GRAB	SURFACE	P	662	66658	.2.	
101 -	17- 53-	1- 16	SOIL	SQUARE 3-W 27	GRAB	SURFACE	P	663	66659	.2.	
102 -	17- 54-	1- 16	SOIL	SQUARE 3-W 28	GRAB	SURFACE	P	664	66660	.2.	
103 -	17- 55-	1- 16	SOIL	SQUARE 3-W 29	GRAB	SURFACE	P	665	66661	.2.	
104 -	17- 56-	1- 16	SOIL	SQUARE 3-W 30	GRAB	SURFACE	P	666	66662	.2.	
105 -	17- 57-	1- 17	SOIL	SQUARE 4-E 31	GRAB	SURFACE	Q	667	66663	.2.	
106 -	17- 58-	1- 16	SOIL	SQUARE 4-E 32	GRAB	SURFACE	P	668	66664 DUPL	.2.	67450
107 -	17- 59-	1- 17	SOIL	SQUARE 4-N 33	GRAB	SURFACE	Q	669	66665	.2.	
108 -	17- 60-	1- 16	SOIL	SQUARE 4-N 34	GRAB	SURFACE	P	670	66666	.2.	
109 -	17- 61-	1- 17	SOIL	SQUARE 4-N 35	GRAB	SURFACE	Q	671	66667	.2.	
110 -	17- 62-	1- 16	SOIL		GRAB		P	672	66668	.2.	
111 -	17- 63-	1- 16	SOIL		GRAB		P	673	66669	.2.	
112 -	17- 64-	1- 16	SOIL		GRAB		P	674	66670	.2.	
113 -											
114 -											
115 -											
116 -											
117 -											
118 -											
119 -	22- 3-	3- 18	SEDIMENT	STREAM - 1	GRAB	0-1 FT	R	675	66671	.2.	
120 -	22- 4-	3- 18	SEDIMENT	STREAM - 2	GRAB	0-1 FT	R	676	66672	.2.	

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GROUP: #8 SITE: 22:OLD REFUGE SHOP

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121 -	22- 5- 3- 18	SEDIMENT	STREAM - 3	GRAB	0-1 FT	R	677	66673	.2.	
122 -	22- 6- 3- 18	SEDIMENT	STREAM - 4	GRAB	0-1 FT	R	678	66674	.2.	
123 -	22- 7- 3- 18	SEDIMENT	STREAM - 5	GRAB	0-1 FT	R	679	66675	.2.	
124 -										
125 -										
126 -										
127 -			GROUP: #10 SITE: 29:FIRE STATION LANDFILL							
128 -										
129 -										
130 -	29- 12- 1- 19	SOIL	EAST FACE 5	GRAB	0-1 FT	S	680	66676 DUPL	.2.	67451
131 -	29- 13- 1- 19	SOIL	EAST FACE 5	GRAB	3 FT	S	681	66677	.2.	
132 -	29- 14- 1- 19	SOIL	EAST FACE 6	GRAB	0-1 FT	S	682	66678 SPKE	.2.	67452
133 -	29- 15- 1- 19	SOIL	EAST FACE 7	GRAB	0-1 FT	S	683	66679	.2.	
134 -	29- 16- 1- 19	SOIL	EAST FACE 8	GRAB	0-1 FT	S	684	66680 FMS	.2.	
135 -	29- 17- 1- 19	SOIL	EAST FACE 9	GRAB	0-1 FT	S	685	66681	.2.	
136 -	29- 18- 1- 19	SOIL	EAST FACE 10	GRAB	0-1 FT	S	686	66682	.2.	
137 -	29- 19- 1- 19	SOIL	EAST FACE 10	GRAB	3 FT	S	687	66683	.2.	
138 -	29- 20- 1- 19	SOIL	EAST FACE 11	GRAB	0-1 FT	S	688	66684	.2.	
139 -	29- 21- 1- 19	SOIL	EAST FACE 12	GRAB	0-1 FT	S	689	66685	.2.	
140 -	29- 22- 1- 19	SOIL	EAST FACE 13	GRAB	0-1 FT	S	690	66686	.2.	
141 -	29- 23- 1- 19	SOIL	EAST FACE 13	GRAB	3 FT	S	691	66687	.2.	
142 -	29- 24- 1- 19	SOIL	EAST FACE 14	GRAB	0-1 FT	S	692	66688	.2.	
143 -										
144 -										
145 -										
146 -			GROUP: #11 SITE: 32:AREA 9 LANDFILL							
147 -										
148 -										
149 -	32- 67- 3- 2	SEDIMENT	GRID 1 - 1	SINGLE SAMPLE	SURFACE	B	693	66689 DUPL	.2.	67453
150 -	32- 68- 3- 2	SEDIMENT	GRID 1 - 2	SINGLE SAMPLE	SURFACE	B	694	66690 FMS	.2.	
151 -	32- 69- 3- 2	SEDIMENT	GRID 1 - 3	SINGLE SAMPLE	SURFACE	B	695	66691 SPKE	.2.	67453
152 -	32- 70- 3- 2	SEDIMENT	GRID 1 - 4	SINGLE SAMPLE	SURFACE	B	696	66692	.2.	
153 -	32- 71- 3- 2	SEDIMENT	GRID 1 - 5	SINGLE SAMPLE	SURFACE	B	697	66693	.2.	
154 -	32- 72- 3- 2	SEDIMENT	GRID 2 - 1	SINGLE SAMPLE	SURFACE	B	698	66694	.2.	
155 -	32- 73- 3- 2	SEDIMENT	GRID 2 - 2	SINGLE SAMPLE	SURFACE	B	699	66695	.2.	
156 -	32- 74- 3- 2	SEDIMENT	GRID 2 - 3	SINGLE SAMPLE	SURFACE	B	700	66696	.2.	
157 -	32- 75- 3- 2	SEDIMENT	GRID 2 - 4	SINGLE SAMPLE	SURFACE	B	701	66697 FMS	.2.	
158 -	32- 76- 3- 2	SEDIMENT	GRID 2 - 5	SINGLE SAMPLE	SURFACE	B	702	66698 DUPL	.2.	67455
159 -	32- 77- 3- 2	SEDIMENT	GRID 3 - 1	SINGLE SAMPLE	SURFACE	B	703	66699	.2.	
160 -	32- 78- 3- 2	SEDIMENT	GRID 3 - 2	SINGLE SAMPLE	SURFACE	B	704	66700	.2.	

161 -	32- 79-	3-	2 SEDIMENT	GRID 3 - 3	SINGLE SAMPLE	SURFACE	B	705	66701	.2.	
162 -	32- 80-	3-	2 SEDIMENT	GRID 3 - 4	SINGLE SAMPLE	SURFACE	B	706	66702	.2.	
163 -	32- 81-	3-	2 SEDIMENT	GRID 3 - 5	SINGLE SAMPLE	SURFACE	B	707	66703	.2.	
164 -	32- 82-	3-	2 SEDIMENT	GRID 4 - 1	SINGLE SAMPLE	SURFACE	B	708	66704	.2.	
165 -	32- 83-	3-	2 SEDIMENT	GRID 4 - 2	SINGLE SAMPLE	SURFACE	B	709	66705	.2.	
166 -	32- 84-	3-	2 SEDIMENT	GRID 4 - 3	SINGLE SAMPLE	SURFACE	B	710	66706 DUPL	.2.	67456
167 -	32- 85-	3-	2 SEDIMENT	GRID 4 - 4	SINGLE SAMPLE	SURFACE	B	711	66707	.2.	
168 -	32- 86-	3-	2 SEDIMENT	GRID 4 - 5	SINGLE SAMPLE	SURFACE	B	712	66708	.2.	
169 -	32- 87-	3-	2 SEDIMENT	GRID 5 - 1	SINGLE SAMPLE	SURFACE	B	713	66709	.2.	
170 -	32- 88-	3-	2 SEDIMENT	GRID 5 - 2	SINGLE SAMPLE	SURFACE	B	714	66710	.2.	
171 -	32- 89-	3-	2 SEDIMENT	GRID 5 - 3	SINGLE SAMPLE	SURFACE	B	715	66711	.2.	
172 -	32- 90-	3-	2 SEDIMENT	GRID 5 - 4	SINGLE SAMPLE	SURFACE	B	716	66712	.2.	
173 -	32- 91-	3-	2 SEDIMENT	GRID 5 - 5	SINGLE SAMPLE	SURFACE	B	717	66713	.2.	
174 -	32- 92-	3-	2 SEDIMENT	GRID 6 - 1	SINGLE SAMPLE	SURFACE	B	718	66714 SPKE	.2.	67457
175 -	32- 93-	3-	2 SEDIMENT	GRID 6 - 2	SINGLE SAMPLE	SURFACE	B	719	66715	.2.	
176 -	32- 94-	3-	2 SEDIMENT	GRID 6 - 3	SINGLE SAMPLE	SURFACE	B	720	66716	.2.	
177 -	32- 95-	3-	2 SEDIMENT	GRID 6 - 4	SINGLE SAMPLE	SURFACE	B	721	66717	.2.	
178 -	32- 96-	3-	2 SEDIMENT	GRID 6 - 5	SINGLE SAMPLE	SURFACE	B	722	66718	.2.	
179 -	32- 97-	3-	2 SEDIMENT	BAY SE - 1	SINGLE SAMPLE	SURFACE	B	723	66719 FWS	.2.	
180 -	32- 98-	3-	2 SEDIMENT	BAY SE - 2	SINGLE SAMPLE	SURFACE	B	724	66720 DUPL	.2.	67458
181 -	32- 99-	3-	2 SEDIMENT	BAY SE - 3	SINGLE SAMPLE	SURFACE	B	725	66721	.2.	
182 -	32-100-	3-	2 SEDIMENT	BAY SE - 4	SINGLE SAMPLE	SURFACE	B	726	66722	.2.	
183 -	32-101-	3-	2 SEDIMENT	BAY SE - 5	SINGLE SAMPLE	SURFACE	B	727	66723	.2.	
184 -	32-102-	3-	2 SEDIMENT	BAY SE - 6	SINGLE SAMPLE	SURFACE	B	728	66724	.2.	
185 -	32-103-	3-	2 SEDIMENT	BAY SE - 7	SINGLE SAMPLE	SURFACE	B	729	66725	.2.	
186 -	32-104-	3-	2 SEDIMENT	BAY SE - 8	SINGLE SAMPLE	SURFACE	B	730	66726	.2.	
187 -	32-105-	3-	2 SEDIMENT	BAY SE - 9	SINGLE SAMPLE	SURFACE	B	731	66727	.2.	
188 -	32-106-	3-	2 SEDIMENT	BAY SE - 10	SINGLE SAMPLE	SURFACE	B	732	66728	.2.	
189 -	32-107-	3-	2 SEDIMENT	BAY SE - 11	SINGLE SAMPLE	SURFACE	B	733	66729 SPKE	.2.	67459
190 -	32-108-	3-	2 SEDIMENT	BAY SE - 12	SINGLE SAMPLE	SURFACE	B	734	66730	.2.	
191 -	32-109-	3-	2 SEDIMENT	BAY SW - 1	SINGLE SAMPLE	SURFACE	B	735	66731	.2.	
192 -	32-110-	3-	2 SEDIMENT	BAY SW - 2	SINGLE SAMPLE	SURFACE	B	736	66732	.2.	
193 -	32-111-	3-	2 SEDIMENT	BAY SW - 3	SINGLE SAMPLE	SURFACE	B	737	66733	.2.	
194 -	32-112-	3-	2 SEDIMENT	BAY SW - 4	SINGLE SAMPLE	SURFACE	B	738	66734 DUPL	.2.	67460
195 -	32-113-	3-	2 SEDIMENT	BAY SW - 5	SINGLE SAMPLE	SURFACE	B	739	66735	.2.	
196 -	32-114-	3-	2 SEDIMENT	BAY MIDDLE - 1	SINGLE SAMPLE	SURFACE	B	740	66736	.2.	
197 -	32-115-	3-	2 SEDIMENT	BAY MIDDLE - 2	SINGLE SAMPLE	SURFACE	B	741	66737	.2.	
198 -	32-116-	3-	2 SEDIMENT	BAY MIDDLE - 3	SINGLE SAMPLE	SURFACE	B	742	66738	.2.	
199 -	32-117-	3-	2 SEDIMENT	BAY NORTH - 1	SINGLE SAMPLE	SURFACE	B	743	66739	.2.	
200 -	32-118-	3-	2 SEDIMENT	BAY NORTH - 2	SINGLE SAMPLE	SURFACE	B	744	66740	.2.	



201 -	32-119-	3-	2 SEDIMENT	BAY NORTH	- 3	SINGLE SAMPLE	SURFACE	B	745	66741		.2.	
202 -	32-120-	3-	2 SEDIMENT	BAY NORTH	- 4	SINGLE SAMPLE	SURFACE	B	746	66742		.2.	
203 -	32-121-	3-	2 SEDIMENT	BAY NORTH	- 5	SINGLE SAMPLE	SURFACE	B	747	66743		.2.	
204 -	32-122-	3-	2 SEDIMENT	BAY NORTH	- 6	SINGLE SAMPLE	SURFACE	B	748	66744		.2.	
205 -	32-123-	3-	2 SEDIMENT	BAY NORTH	- 7	SINGLE SAMPLE	SURFACE	B	749	66745		.2.	
206 -													
207 -													
208 -													
209 -													
210 -													
211 -													
212 -	33-189-	1-	2 SOIL LOC.	103 - 1-1-	25	CORE VERTICAL	0-1 FOOT	B	400	11775	FWS	.2.	
213 -	33-190-	1-	2 SOIL LOC.	103 - 1-1-	25	CORE VERTICAL	1-2 FOOT	B	401	11776		.2.	
214 -	33-191-	1-	2 SOIL LOC.	103 - 1-1-	25	CORE VERTICAL	2-3 FOOT	B	402	11777		.2.	
215 -	33-192-	1-	2 SOIL LOC.	104 - 1-1-	25	CORE VERTICAL	0-1 FOOT	B	403	11778		.2.	
216 -	33-193-	1-	2 SOIL LOC.	104 - 1-1-	25	CORE VERTICAL	1-2 FOOT	B	404	11779	DUPL	.2.	19216
217 -	33-194-	1-	2 SOIL LOC.	104 - 1-1-	25	CORE VERTICAL	2-3 FOOT	B	405	11780		.2.	
218 -	33-195-	1-	2 SOIL LOC.	105 - 1-1-	25	CORE VERTICAL	0-1 FOOT	B	406	11781	DUPL	.2.	67461
219 -	33-196-	1-	2 SOIL LOC.	105 - 1-1-	25	CORE VERTICAL	1-2 FOOT	B	407	11782		.2.	
220 -	33-197-	1-	2 SOIL LOC.	105 - 1-1-	25	CORE VERTICAL	2-3 FOOT	B	408	11783		.2.	
221 -	33-198-	3-	2 SEDIMENT LOC.	106 - NW.DRNG		CORE VERTICAL	0-1 FOOT	B	409	11784		.2.	
222 -	33-199-	3-	2 SEDIMENT LOC.	106 - NW.DRNG		CORE VERTICAL	1-2 FOOT	B	410	11785		.2.	
223 -	33-200-	3-	2 SEDIMENT LOC.	106 - NW.DRNG		CORE VERTICAL	2-3 FOOT	B	411	11786		.2.	
224 -	33-201-	1-	2 SOIL LOC.	107 - NW.DRNG		SURFACE COMP.	0-1 FOOT	B	412	11787		.2.	
225 -	33-202-	3-	2 SEDIMENT LOC.	108 - NW.DRNG		CORE VERTICAL	0-1 FOOT	B	750	66746	DUPL	.2.	67462
226 -	33-203-	3-	2 SEDIMENT LOC.	108 - NW.DRNG		CORE VERTICAL	1-2 FOOT	B	751	66747		.2.	
227 -	33-204-	3-	2 SEDIMENT LOC.	108 - NW.DRNG		CORE VERTICAL	2-3 FOOT	B	752	66748		.2.	
228 -	33-205-	1-	2 SOIL LOC.	109 - NW.DRNG		SURFACE COMP.	0-1 FOOT	B	753	66749	DUPL	.2.	67463
229 -	33-206-	1-	2 SOIL LOC.	110 - NW.DRNG		SURFACE COMP.	0-1 FOOT	B	754	66750	FWS	.2.	
230 -	33-207-	3-	2 SEDIMENT LOC.	111 - NW.DRNG		CORE VERTICAL	0-1 FOOT	B	755	66751	SPKE	.2.	67464
231 -	33-208-	3-	2 SEDIMENT LOC.	111 - NW.DRNG		CORE VERTICAL	1-2 FOOT	B	756	66752		.2.	
232 -	33-209-	3-	2 SEDIMENT LOC.	111 - NW.DRNG		CORE VERTICAL	2-3 FOOT	B	757	66753		.2.	
233 -	33-210-	1-	2 SOIL LOC.	112 - NW.DRNG		SURFACE COMP.	0-1 FOOT	B	758	66754		.2.	
234 -	33-211-	1-	2 SOIL LOC.	113 - NW.DRNG		SURFACE COMP.	0-1 FOOT	B	759	66755		.2.	
235 -	33-212-	3-	2 SEDIMENT LOC.	114 - NW.DRNG		CORE VERTICAL	0-1 FOOT	B	760	66756		.2.	
236 -	33-213-	3-	2 SEDIMENT LOC.	114 - NW.DRNG		CORE VERTICAL	1-2 FOOT	B	761	66757	DUPL	.2.	67476
237 -	33-214-	3-	2 SEDIMENT LOC.	114 - NW.DRNG		CORE VERTICAL	2-3 FOOT	B	762	66758		.2.	
238 -	33-215-	1-	2 SOIL LOC.	115 - NW.DRNG		SURFACE COMP.	0-1 FOOT	B	763	66759		.2.	
239 -	33-216-	3-	2 SEDIMENT LOC.	116 - NW.DRNG		CORE VERTICAL	0-1 FOOT	B	764	66760		.2.	
240 -	33-217-	3-	2 SEDIMENT LOC.	116 - NW.DRNG		CORE VERTICAL	1-2 FOOT	B	765	66761		.2.	

241 -	33-218-	3- 2	SEDIMENT LOC. 116 -MM.DANG CORE VERTICAL	2-3 FOOT	B	766	66762		.2.	
242 -	33-219-	1- 2	SOIL LOC. 117 - I-1- 23 CORE VERTICAL	3-4 FOOT	B	767	66763	FWS	.2.	
243 -	33-220-	1- 2	SOIL LOC. 117 - I-1- 23 CORE VERTICAL	4-5 FOOT	B	768	66764		.2.	
244 -	33-221-	1- 2	SOIL LOC. 117 - I-1- 23 CORE VERTICAL	5-6 FOOT	B	769	66765	DUPL	.2.	67477
245 -	33-222-	1- 2	SOIL LOC. 118 - I-1- 23 CORE VERTICAL	3-4 FOOT	B	770	66766	DUPL	.2.	67465
246 -	33-223-	1- 2	SOIL LOC. 118 - I-1- 23 CORE VERTICAL	4-5 FOOT	B	771	66767		.2.	
247 -	33-224-	1- 2	SOIL LOC. 118 - I-1- 23 CORE VERTICAL	5-6 FOOT	B	772	66768		.2.	
248 -	33-225-	1- 2	SOIL LOC. 119 - I-1- 23 CORE VERTICAL	3-4 FOOT	B	773	66769		.2.	
249 -	33-226-	1- 2	SOIL LOC. 119 - I-1- 23 CORE VERTICAL	4-5 FOOT	B	774	66770		.2.	
250 -	33-227-	1- 2	SOIL LOC. 119 - I-1- 23 CORE VERTICAL	5-6 FOOT	B	775	66771		.2.	
251 -	33-228-	1- 2	SOIL LOC. 120 - I-1- 23 CORE VERTICAL	3-4 FOOT	B	776	66772		.2.	
252 -	33-229-	1- 2	SOIL LOC. 120 - I-1- 23 CORE VERTICAL	4-5 FOOT	B	777	66773	SPKE	.2.	19283
253 -	33-230-	1- 2	SOIL LOC. 120 - I-1- 23 CORE VERTICAL	5-6 FOOT	B	778	66774		.2.	
254 -	33-231-	1- 2	SOIL LOC. 121 - I-1- 23 CORE VERTICAL	0-1 FOOT	B	779	66775		.2.	
255 -	33-232-	1- 2	SOIL LOC. 121 - I-1- 23 CORE VERTICAL	1-2 FOOT	B	780	66776		.2.	
256 -	33-233-	1- 2	SOIL LOC. 121 - I-1- 23 CORE VERTICAL	2-3 FOOT	B	781	66777		.2.	
257 -	33-234-	1- 2	SOIL LOC. 122 - I-1- 23 CORE VERTICAL	0-1 FOOT	B	782	66778		.2.	
258 -	33-235-	1- 2	SOIL LOC. 122 - I-1- 23 CORE VERTICAL	1-2 FOOT	B	783	66779		.2.	
259 -	33-236-	1- 2	SOIL LOC. 122 - I-1- 23 CORE VERTICAL	2-3 FOOT	B	784	66780		.2.	
260 -	33-237-	1- 2	SOIL LOC. 123 - I-1- 23 CORE VERTICAL	0-1 FOOT	B	785	66781		.2.	
261 -	33-238-	1- 2	SOIL LOC. 123 - I-1- 23 CORE VERTICAL	1-2 FOOT	B	786	66782		.2.	
262 -	33-239-	1- 2	SOIL LOC. 123 - I-1- 23 CORE VERTICAL	2-3 FOOT	B	787	66783		.2.	
263 -	33-240-	1- 2	SOIL LOC. 124 - I-1- 23 CORE VERTICAL	0-1 FOOT	B	788	66784	SPKE	.2.	67466
264 -	33-241-	1- 2	SOIL LOC. 124 - I-1- 23 CORE VERTICAL	1-2 FOOT	B	789	66785		.2.	
265 -	33-242-	1- 2	SOIL LOC. 124 - I-1- 23 CORE VERTICAL	2-3 FOOT	B	790	66786		.2.	
266 -	33-243-	1- 2	SOIL LOC. 125 - I-1- 23 CORE VERTICAL	0-1 FOOT	B	791	66787		.2.	
267 -	33-244-	1- 2	SOIL LOC. 125 - I-1- 23 CORE VERTICAL	1-2 FOOT	B	792	66788		.2.	
268 -	33-245-	1- 2	SOIL LOC. 125 - I-1- 23 CORE VERTICAL	2-3 FOOT	B	793	66789		.2.	
269 -	33-246-	1- 2	SOIL LOC. 126 - I-1- 23 CORE VERTICAL	0-1 FOOT	B	794	66790		.2.	
270 -	33-247-	1- 2	SOIL LOC. 126 - I-1- 23 CORE VERTICAL	1-2 FOOT	B	795	66791	DUPL	.2.	64492
271 -	33-248-	1- 2	SOIL LOC. 126 - I-1- 23 CORE VERTICAL	2-3 FOOT	B	796	66792		.2.	
272 -	33-249-	1- 2	SOIL LOC. 127 - I-1- 23 CORE VERTICAL	0-1 FOOT	B	797	66793		.2.	
273 -	33-250-	1- 2	SOIL LOC. 127 - I-1- 23 CORE VERTICAL	1-2 FOOT	B	798	66794		.2.	
274 -	33-251-	1- 2	SOIL LOC. 127 - I-1- 23 CORE VERTICAL	2-3 FOOT	B	799	66795		.2.	
275 -	33-252-	1- 2	SOIL LOC. 128 - I-1- 23 CORE VERTICAL	0-1 FOOT	B	800	66796		.2.	
276 -	33-253-	1- 2	SOIL LOC. 128 - I-1- 23 CORE VERTICAL	1-2 FOOT	B	801	66797		.2.	
277 -	33-254-	1- 2	SOIL LOC. 128 - I-1- 23 CORE VERTICAL	2-3 FOOT	B	802	66798		.2.	
278 -	33-255-	1- 2	SOIL LOC. 129 - I-1- 23 CORE VERTICAL	0-1 FOOT	B	803	66799		.2.	
279 -	33-256-	1- 2	SOIL LOC. 129 - I-1- 23 CORE VERTICAL	1-2 FOOT	B	842	66403		.2.	
280 -	33-257-	1- 2	SOIL LOC. 129 - I-1- 23 CORE VERTICAL	2-3 FOOT	B	843	66404		.2.	

281 -	33-258-	1- 2	SOIL LOC. 130 - I-1- 23	CORE VERTICAL	0-1 FOOT	B	844	66405		.2.	
282 -	33-259-	1- 2	SOIL LOC. 130 - I-1- 23	CORE VERTICAL	1-2 FOOT	B	845	66406		.2.	
283 -	33-260-	1- 2	SOIL LOC. 130 - I-1- 23	CORE VERTICAL	2-3 FOOT	B	846	66407		.2.	
284 -	33-261-	1- 2	SOIL LOC. 131 - I-1- 23	CORE VERTICAL	0-1 FOOT	B	847	66408		.2.	
285 -	33-262-	1- 2	SOIL LOC. 131 - I-1- 23	CORE VERTICAL	1-2 FOOT	B	848	66409		.2.	
286 -	33-263-	1- 2	SOIL LOC. 131 - I-1- 23	CORE VERTICAL	2-3 FOOT	B	849	66410		.2.	
287 -	33-264-	1- 2	SOIL LOC. 132 - I-1- 5	CORE VERTICAL	0-1 FOOT	B	850	66411	FWS	.2.	
288 -	33-265-	1- 2	SOIL LOC. 132 - I-1- 5	CORE VERTICAL	1-2 FOOT	B	851	66412		.2.	
289 -	33-266-	1- 2	SOIL LOC. 132 - I-1- 5	CORE VERTICAL	2-3 FOOT	B	852	66413		.2.	
290 -	33-267-	1- 2	SOIL LOC. 133 - I-1- 5	CORE VERTICAL	0-1 FOOT	B	853	66414		.2.	
291 -	33-268-	1- 2	SOIL LOC. 133 - I-1- 5	CORE VERTICAL	1-2 FOOT	B	854	66415		.2.	
292 -	33-269-	1- 2	SOIL LOC. 133 - I-1- 5	CORE VERTICAL	2-3 FOOT	B	855	66416		.2.	
293 -	33-270-	1- 2	SOIL LOC. 134 - I-1- 5	CORE VERTICAL	3-4 FOOT	B	856	66417	DUPL	.2.	67467
294 -	33-271-	1- 2	SOIL LOC. 134 - I-1- 5	CORE VERTICAL	4-5 FOOT	B	857	66418		.2.	
295 -	33-272-	1- 2	SOIL LOC. 134 - I-1- 5	CORE VERTICAL	5-6 FOOT	B	858	66419		.2.	
296 -	33-273-	1- 2	SOIL LOC. 135 - I-1- 5	CORE VERTICAL	0-1 FOOT	B	859	66420		.2.	
297 -	33-274-	1- 2	SOIL LOC. 135 - I-1- 5	CORE VERTICAL	1-2 FOOT	B	860	66421		.2.	
298 -	33-275-	1- 2	SOIL LOC. 135 - I-1- 5	CORE VERTICAL	2-3 FOOT	B	861	66422		.2.	
299 -	33-276-	1- 2	SOIL LOC. 136 - I-1- 5	CORE VERTICAL	0-1 FOOT	B	862	66423		.2.	
300 -	33-277-	1- 2	SOIL LOC. 136 - I-1- 5	CORE VERTICAL	1-2 FOOT	B	863	66424	SPKE	.2.	19284
301 -	33-278-	1- 2	SOIL LOC. 136 - I-1- 5	CORE VERTICAL	2-3 FOOT	B	864	66425		.2.	
302 -	33-279-	1- 2	SOIL LOC. 137 - I-1- 5	CORE VERTICAL	3-4 FOOT	B	865	66426		.2.	
303 -	33-280-	1- 2	SOIL LOC. 137 - I-1- 5	CORE VERTICAL	4-5 FOOT	B	866	66427		.2.	
304 -	33-281-	1- 2	SOIL LOC. 137 - I-1- 5	CORE VERTICAL	5-6 FOOT	B	867	66428		.2.	
305 -	33-282-	1- 2	SOIL LOC. 138 - I-1- 5	CORE VERTICAL	0-1 FOOT	B	868	66429		.2.	
306 -	33-283-	1- 2	SOIL LOC. 138 - I-1- 5	CORE VERTICAL	1-2 FOOT	B	869	66430		.2.	
307 -	33-284-	1- 2	SOIL LOC. 138 - I-1- 5	CORE VERTICAL	2-3 FOOT	B	870	66431		.2.	
308 -	33-285-	1- 2	SOIL LOC. 139 - I-1- 5	CORE VERTICAL	0-1 FOOT	B	871	66432	SPKE	.2.	67468
309 -	33-286-	1- 2	SOIL LOC. 139 - I-1- 5	CORE VERTICAL	1-2 FOOT	B	872	66433		.2.	
310 -	33-287-	1- 2	SOIL LOC. 139 - I-1- 5	CORE VERTICAL	2-3 FOOT	B	873	66434		.2.	
311 -	33-288-	1- 2	SOIL LOC. 140 - I-1- 5	CORE VERTICAL	0-1 FOOT	B	874	66435		.2.	
312 -	33-289-	1- 2	SOIL LOC. 140 - I-1- 5	CORE VERTICAL	1-2 FOOT	B	875	66436	DUPL	.2.	64493
313 -	33-290-	1- 2	SOIL LOC. 140 - I-1- 5	CORE VERTICAL	2-3 FOOT	B	876	66437		.2.	
314 -	33-291-	1- 2	SOIL LOC. 141 - NW.DRNG	CORE VERTICAL	0-1 FOOT	B	877	66438	FWS	.2.	
315 -	33-292-	1- 2	SOIL LOC. 141 - NW.DRNG	CORE VERTICAL	1-2 FOOT	B	878	66439		.2.	
316 -	33-293-	1- 2	SOIL LOC. 141 - NW.DRNG	CORE VERTICAL	2-3 FOOT	B	879	66440		.2.	
317 -	33-294-	1- 2	SOIL LOC. 142 - NW.DRNG	CORE VERTICAL	0-1 FOOT	B	880	66441		.2.	
318 -	33-295-	1- 2	SOIL LOC. 142 - NW.DRNG	CORE VERTICAL	1-2 FOOT	B	881	66442		.2.	
319 -	33-296-	1- 2	SOIL LOC. 142 - NW.DRNG	CORE VERTICAL	2-3 FOOT	B	882	66443		.2.	
320 -	33-297-	1- 2	SOIL LOC. 143 - NW.DRNG	SURFACE COMP.	0-1 FOOT	B	883	66444		.2.	

321 -	33-298-	3-	2	SEDIMENT LOC. 144 -NW.DRNG CORE VERTICAL	0-1 FOOT	B	884	66445	.2.	
322 -	33-299-	3-	2	SEDIMENT LOC. 144 -NW.DRNG CORE VERTICAL	1-2 FOOT	B	885	66446 DUPL	.2.	64494
323 -	33-300-	3-	2	SEDIMENT LOC. 144 -NW.DRNG CORE VERTICAL	2-3 FOOT	B	886	66447	.2.	
324 -	33-301-	1-	2	SOIL LOC. 145 - NW.DRNG SURFACE COMP.	0-1 FOOT	B	887	66448	.2.	
325 -	33-302-	1-	2	SOIL LOC. 146 - NW.DRNG SURFACE COMP.	0-1 FOOT	B	888	66449	.2.	
326 -	33-303-	3-	2	SEDIMENT LOC. 147 -NW.DRNG CORE VERTICAL	0-1 FOOT	B	889	66450	.2.	
327 -	33-304-	3-	2	SEDIMENT LOC. 147 -NW.DRNG CORE VERTICAL	1-2 FOOT	B	890	66451	.2.	
328 -	33-305-	3-	2	SEDIMENT LOC. 147 -NW.DRNG CORE VERTICAL	2-3 FOOT	B	891	66452	.2.	
329 -	33-306-	1-	2	SOIL LOC. 148 - NW.DRNG SURFACE COMP.	0-1 FOOT	B	892	66453	.2.	
330 -	33-307-	1-	2	SOIL LOC. 149 - NW.DRNG SURFACE COMP.	0-1 FOOT	B	893	66454	.2.	
331 -	33-308-	3-	2	SEDIMENT LOC. 150 -NW.DRNG CORE VERTICAL	0-1 FOOT	B	894	66455 DUPL	.2.	67469
332 -	33-309-	3-	2	SEDIMENT LOC. 150 -NW.DRNG CORE VERTICAL	1-2 FOOT	B	895	66456	.2.	
333 -	33-310-	3-	2	SEDIMENT LOC. 150 -NW.DRNG CORE VERTICAL	2-3 FOOT	B	896	66457	.2.	
334 -	33-311-	1-	2	SOIL LOC. 151 - NW.DRNG SURFACE COMP.	0-1 FOOT	B	897	66458 FWS	.2.	
335 -	33-312-	1-	2	SOIL LOC. 152 - NW.DRNG SURFACE COMP.	0-1 FOOT	B	898	66459	.2.	
336 -	33-313-	3-	2	SEDIMENT LOC. 153 -NW.DRNG CORE VERTICAL	0-1 FOOT	B	899	66460	.2.	
337 -	33-314-	3-	2	SEDIMENT LOC. 153 -NW.DRNG CORE VERTICAL	1-2 FOOT	B	900	66461 DUPL	.2.	64495
338 -	33-315-	3-	2	SEDIMENT LOC. 153 -NW.DRNG CORE VERTICAL	2-3 FOOT	B	901	66462	.2.	
339 -	33-316-	1-	2	SOIL LOC. 154 - NW.DRNG SURFACE COMP.	0-1 FOOT	B	902	66463	.2.	
340 -	33-317-	1-	2	SOIL LOC. 155 - NW.DRNG SURFACE COMP.	0-1 FOOT	B	903	66464	.2.	
341 -	33-318-	3-	2	SEDIMENT LOC. 156 -NW.DRNG CORE VERTICAL	0-1 FOOT	B	904	66465	.2.	
342 -	33-319-	3-	2	SEDIMENT LOC. 156 -NW.DRNG CORE VERTICAL	1-2 FOOT	B	905	66466	.2.	
343 -	33-320-	3-	2	SEDIMENT LOC. 156 -NW.DRNG CORE VERTICAL	2-3 FOOT	B	906	66467	.2.	
344 -	33-321-	1-	2	SOIL LOC. 157 -NW.DRNG SURFACE COMP.	0-1 FOOT	B	907	66468	.2.	
345 -	33-322-	3-	2	SEDIMENT LOC. 158 -NW.DRNG CORE VERTICAL	0-1 FOOT	B	908	66469	.2.	
346 -	33-323-	3-	2	SEDIMENT LOC. 158 -NW.DRNG CORE VERTICAL	1-2 FOOT	B	909	66470 SPKE	.2.	64497
347 -	33-324-	3-	2	SEDIMENT LOC. 158 -NW.DRNG CORE VERTICAL	2-3 FOOT	B	910	66471	.2.	
348 -	33-325-	3-	2	SEDIMENT LOC. 159 -NW.DRNG CORE VERTICAL	0-1 FOOT	B	911	66472	.2.	
349 -	33-326-	3-	2	SEDIMENT LOC. 159 -NW.DRNG CORE VERTICAL	1-2 FOOT	B	912	66473	.2.	
350 -	33-327-	3-	2	SEDIMENT LOC. 159 -NW.DRNG CORE VERTICAL	2-3 FOOT	B	913	66474	.2.	
351 -	33-328-	3-	2	SEDIMENT LOC. 160 -NW.DRNG CORE VERTICAL	0-1 FOOT	B	914	66475	.2.	
352 -	33-329-	3-	2	SEDIMENT LOC. 160 -NW.DRNG CORE VERTICAL	1-2 FOOT	B	915	66476	.2.	
353 -	33-330-	3-	2	SEDIMENT LOC. 160 -NW.DRNG CORE VERTICAL	2-3 FOOT	B	916	66477	.2.	
354 -	33-331-	3-	2	SEDIMENT LOC. 161 -NW.DRNG CORE VERTICAL	0-1 FOOT	B	917	66478	.2.	
355 -	33-332-	3-	2	SEDIMENT LOC. 161 -NW.DRNG CORE VERTICAL	1-2 FOOT	B	918	66479	.2.	
356 -	33-333-	3-	2	SEDIMENT LOC. 161 -NW.DRNG CORE VERTICAL	2-3 FOOT	B	919	66480	.2.	
357 -	33-334-	3-	2	SEDIMENT LOC. 162 -NW.DRNG CORE VERTICAL	0-1 FOOT	B	920	66481	.2.	
358 -	33-335-	3-	2	SEDIMENT LOC. 162 -NW.DRNG CORE VERTICAL	1-2 FOOT	B	921	64485 DUPL	.2.	64496
359 -	33-336-	3-	2	SEDIMENT LOC. 162 -NW.DRNG CORE VERTICAL	2-3 FOOT	B	922	64486	.2.	
360 -	33-337-	3-	2	SEDIMENT LOC. 163 -NW.DRNG CORE VERTICAL	0-1 FOOT	B	923	64487	.2.	

361 -	33-338-	3-	2	SEDIMENT LOC. 163 -NM.DANG CORE VERTICAL	1-2 FOOT	B	924	64488	.2.
362 -	33-339-	3-	2	SEDIMENT LOC. 163 -NM.DANG CORE VERTICAL	2-3 FOOT	B	925	64489	.2.
363 -	33-340-	1-	2	SOIL -SPARE		B	926	64490	.
364 -	33-341-	1-	2	SOIL -SPARE		B	927	64491	.
365 -	33-342-	1-	2	SOIL -SPARE		B	934	64498	.
366 -	33-343-	1-	2	SOIL -SPARE		B	935	64499	.

367 -

368 -

369 -

370 -

371 -

372 -

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 GROUP: #14 SITE: 40:REPLICATES
 

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373 -	40- 2-	1-	2	SOIL	REPLCTE 2	B	477	19216 DUPL	.2.	11779
374 -	40- 54-	3-	10	SEDIMENT	REPLCTE 2	J	804	67440 DUPL	.2.	66602
375 -	40- 55-	3-	11	SEDIMENT	REPLCTE 2	K	805	67441 DUPL	.2.	66611
376 -	40- 56-	3-	12	SEDIMENT	REPLCTE 2	L	806	67442 DUPL	.2.	66613
377 -	40- 57-	1-	16	SOIL	REPLCTE 2	P	807	67443 DUPL	.2.	66624
378 -	40- 58-	1-	16	SOIL	REPLCTE 2	P	809	67445 DUPL	.2.	66628
379 -	40- 59-	1-	17	SOIL	REPLCTE 2	Q	810	67446 DUPL	.2.	66636
380 -	40- 60-	1-	17	SOIL	REPLCTE 2	Q	811	67447 DUPL	.2.	66640
381 -	40- 61-	1-	16	SOIL	REPLCTE 2	P	812	67448 DUPL	.2.	66648
382 -	40- 62-	1-	16	SOIL	REPLCTE 2	P	814	67450 DUPL	.2.	66664
383 -	40- 63-	1-	19	SOIL	REPLCTE 2	S	815	67451 DUPL	.2.	66676
384 -	40- 64-	3-	2	SEDIMENT	REPLCTE 2	B	817	67453 DUPL	.2.	66689
385 -	40- 65-	3-	2	SEDIMENT	REPLCTE 2	B	819	67455 DUPL	.2.	66698
386 -	40- 66-	3-	2	SEDIMENT	REPLCTE 2	B	820	67456 DUPL	.2.	66706
387 -	40- 67-	3-	2	SEDIMENT	REPLCTE 2	B	822	67458 DUPL	.2.	66720
388 -	40- 68-	3-	2	SEDIMENT	REPLCTE 2	B	824	67460 DUPL	.2.	66734
389 -	40- 69-	1-	2	SOIL	REPLCTE 2	B	825	67461 DUPL	.2.	11781
390 -	40- 70-	1-	2	SOIL	REPLCTE 2	B	826	67462 DUPL	.2.	66746
391 -	40- 71-	1-	2	SOIL	REPLCTE 2	B	827	67463 DUPL	.2.	66749
392 -	40- 72-	1-	2	SOIL	REPLCTE 2	B	829	67465 DUPL	.2.	66766
393 -	40- 73-	1-	2	SOIL	REPLCTE 2	B	831	67467 DUPL	.2.	66417
394 -	40- 74-	1-	2	SOIL	REPLCTE 2	B	833	67469 DUPL	.2.	66455
395 -	40- 75-	1-	2	SOIL	REPLCTE 2	B	840	67476 DUPL	.2.	66757
396 -	40- 76-	1-	2	SOIL	REPLCTE 2	B	841	67477 DUPL	.2.	66765
397 -	40- 77-	1-	2	SOIL	REPLCTE 2	B	928	64492 DUPL	.2.	66791
398 -	40- 78-	1-	2	SOIL	REPLCTE 2	B	929	64493 DUPL	.2.	66436
399 -	40- 79-	1-	2	SOIL	REPLCTE 2	B	930	64494 DUPL	.2.	66446
400 -	40- 80-	1-	2	SOIL	REPLCTE 2	B	931	64495 DUPL	.2.	66461

401 -	40- 81- 1- 2	SOIL	REPLCTE 2	B	932 64496 DUPL	.2.	64485
402 -							
403 -							
404 -							
405 -	GROUP: #14 SITE: 41: MATRIX SPIKES						
406 -							
407 -							
408 -	41- 22- 1- 2	SOIL	SPIKE ONLY	B	544 19283	.2.	66773
409 -	41- 23- 1- 2	SOIL	SPIKE ONLY	B	545 19284	.2.	66424
410 -	41- 31- 1- 16	SOIL	SPIKE ONLY	P	808 67444 SPKE	.2.	66626
411 -	41- 32- 1- 16	SOIL	SPIKE ONLY	P	813 67449 SPKE	.2.	66656
412 -	41- 33- 1- 19	SOIL	SPIKE ONLY	S	816 67452 SPKE	.2.	66678
413 -	41- 34- 3- 2	SEDIMENT	SPIKE ONLY	B	818 67454 SPKE	.2.	66691
414 -	41- 35- 3- 2	SEDIMENT	SPIKE ONLY	B	821 67457 SPKE	.2.	66714
415 -	41- 36- 3- 2	SEDIMENT	SPIKE ONLY	B	823 67459 SPKE	.2.	66729
416 -	41- 37- 1- 2	SOIL	SPIKE ONLY	B	828 67464 SPKE	.2.	66751
417 -	41- 38- 1- 2	SOIL	SPIKE ONLY	B	830 67466 SPKE	.2.	66784
418 -	41- 39- 1- 2	SOIL	SPIKE ONLY	B	832 67468 SPKE	.2.	66432
419 -	41- 40- 1- 2	SOIL	SPIKE ONLY	B	933 64497 SPKE	.	66470
420 -	41- 41- 1- 2	SOIL -SPARE	SPIKE ONLY	B	936 64500 SPKE	.	
421 -							
422 -							
423 -							
424 -	GROUP: #14 SITE: 42: BLANKS						
425 -							
426 -							
427 -	42- 15- 1- 2	SOIL OB46 LAB	BLANK	B	834 67470 BLNK	.2.	
428 -	42- 16- 1- 2	SOIL OB46 LAB	BLANK	B	835 67471 BLNK	.2.	
429 -	42- 17- 1- 10	SOIL OB46 LAB	BLANK	J	836 67472 BLNK	.2.	
430 -	42- 18- 1- 11	SOIL OB46 LAB	BLANK	K	837 67473 BLNK	.2.	
431 -	42- 19- 1- 16	SOIL OB46 LAB	BLANK	P	838 67474 BLNK	.2.	
432 -	42- 20- 1- 17	SOIL OB46 LAB	BLANK	Q	839 67475 BLNK	.2.	
433 -							
434 -							
435 -							
436 -							
437 -							
438 -							
439 -							
440 -							

## ATTACHMENT 2

### KEY TO RATIONALE FOR SAMPLING DEPTH, LOCATION AND INTERVAL

## ATTACHMENT 2

### REMEDIAL INVESTIGATION/FEASIBILITY STUDY CRAB ORCHARD NATIONAL WILDLIFE REFUGE

#### SPECIFIC RATIONALE FACTORS FOR SELECTING SAMPLES AND ANALYSES FOR PHASE I

#### KEY TO RATIONALE FACTORS CITED IN ATTACHMENT 2 (Used in Sampling - See Attachment 1)

#### ANALYSIS SETS

- A. Analysis Set A (organics screening, metals, cyanide, indicators, explosives, nitrogen and phosphorus) is specified for all sites where few or no previous analytical tests have been conducted. This sequence of analyses was chosen to enable problem identification associated with the range of industries which have operated on the Refuge. The results of organics screening are used to determine sampling locations for resampling and analysis by full CLP protocol (Analysis Set F).
- B. Analysis Set B consists only of PCB analysis. This set is specified only for samples within the Area 9 Building Complex (Site 33) and portions of the Area 9 Landfill (Site 32) where the spatial distribution and limits of PCB contamination are to be determined.
- C. Analysis Set C consists of PCBs, PCDF/PCDD screening, indicators, nitrogen and cation exchange capacity. Set C analyses are specified only for intermediate core sections (top, middle and bottom) within the Area 9 Landfill.

Note: PCDF/PCDD screening in set C has been changed to the full CLP protocol analysis (addition of Set H) during the initial sampling effort. This change was necessary to avoid a second round drilling effort in Phase I (d). This represents a change to the original Work Plan.

- D. Analysis Set D is the same as Analysis Set A, except that PCDF/PCDD screening is also included. One sample (and sometimes 2 or 3) was selected from each site which was anticipated to contain PCBs or organics for PCDF/PCDD screening.
- E. Analysis Set E consists of the Primary and Secondary Drinking Water Standards. These are conducted only on the Phase I water supply samples (Samples 34-1 through 34-5). Phase II Crab Orchard Lake water column samples (Samples 34-6 through 34-15) are to be analyzed for a wider range of parameters which will be selected after evaluation of results from the Phase I sampling effort. However, at least one water column sample will be analyzed for the full Set G suite of parameters, as requested by U.S. EPA. This represents a change to the original Work Plan.



- F. Analysis Set F is conducted on a second round (Phase I (d)) re-sampling for selected locations which were previously analyzed for the Set A suite of analyses. It consists of a second round of organic analyses by full CLP protocol. The rationale for selection of Set F samples is to choose those samples on a given site which show the highest concentrations of organics (by FID screening, PCB, TOC, TOX, or organic nitrogen). The Set F organic CLP analyses will then establish those parameters to be emphasized during the Phase II investigation of extent of contamination.
- G. Analysis Set G is conducted on a second round (Phase I (d)) re-sampling for selected locations which were previously analyzed for the Set A or D suites of analyses by full CLP protocol in addition to PCDF/PCDD analyses by full CLP protocol. The rationale for selection of Set G samples is similar to that for Set F, except that Set G is specified on those sites where PCBs (and hence PCDF/PCDD) are anticipated to be a potential problem.

NOTE: The original Work Plan specified that Priority Pollutant metals analyses by AA Spectroscopy would be conducted for Set F and G samples in addition to the ICP screening for the corresponding Set A and D samples. However, the analytical sensitivity of the ICP analyses has been adequate. Therefore, the AA Spectroscopy metals have been dropped from the Set F and G suites. This represents a change to the original Work Plan.

- H. Analysis Set H was previously full CLP protocol PCDF/PCDD analysis subsequent to screening via Analysis Set C at the Area 9 Landfill. Set H was incorporated into the Set C analyses to avoid second-round drilling at Area 9 (see note following C above). Set H has been redefined to consist of organic analyses by full CLP protocol in addition to PCDF/PCDD analyses by full CLP protocol and other soil characteristics as in Set D. Set H samples are not preceded by a set of screening samples.
- I. Analysis Set I will be conducted on those samples scheduled to be collected during the Phase II sampling effort. Samples for Set I (See Attachment S-4) include groundwater monitoring wells, and Crab Orchard Lake water, sediment and fish. The Set I suite of parameters will depend on the analytical results obtained from Phases Ia, Ib, Ic and Id.

#### SAMPLING DEPTHS

- I. Visual observations indicate the presence of potential contaminants (sludges, debris, etc.) at or near the surface.
- J. Historic information indicated that contaminants may have been disposed or spilled in the general geographic area, but visual observations do not provide guidance to location.

- K. Dispersed contaminants from the potential source area are likely to have accumulated at or near the surface of stream bottoms or drainage swales as a result of sedimentation at points of low flow velocity.
- L. Sampling depths greater than 1 ft were specified because historic information indicated the possibility that source materials may have been buried and covered with soil.
- M. Sampling depths greater than 1 ft were specified to determine if vertical dispersion of near-surface contaminants has occurred.
- N. An upper surface water sample is expected to be representative of the water column as a whole, since stratification is not expected to be significant.
- O. Water column samples are specified for a number of intervals of depth to determine if contaminants are stratified at different concentrations.

#### SAMPLING LOCATIONS

- P. Visual observations indicate the presence of potential contaminants (sludges, debris, etc.) at or near the surface in the location specified for sampling.
- Q. Historic information indicated that contaminants were disposed or spilled in the general geographic area, but visual observations do not provide guidance to location. Therefore, lateral composites were prepared to screen a questionable area. If elevated concentrations of contaminants are found within a compositing area then additional Phase II sampling and analyses may be required to define the lateral and vertical distribution of the contaminated area.
- Q1. Previous sampling and analytical data are available covering this general area. (See Figure 2 of Status Report dated September 11, 1985 for illustrated data for the Area 9 Building Complex, Site 33. Proposed Phase I sampling locations are shown on Figure 1 of that document). The sampling sites were located to permit the evaluation of spatial distributions within previously composited areas. In addition, the sampling locations attempt to define the limits of contaminated areas by the sampling of spots expected to be clean (e.g., outside of ditches and deeper soils underneath contaminated zones).
- R. Dispersed contaminants from a potential source area are likely to have accumulated within streams or drainage swales downgradient or adjacent to the source.
- S. Surface topography indicates that the specified sampling location is downgradient from a number of potential source areas. A broader range of contaminants may be present at this location representing the area as a whole. However, because of differing mobilities

associated with different contaminants, this downgradient sample may not be representative of the distribution of materials in the upgradient sources. In addition, because of dispersion, a downgradient sample is likely to be less concentrated than the source from which it came.

- T. The specified sampling location is believed to be located upgradient of the suspected source area or outside of the dispersion pattern from the suspected source.
- U. The specified sampling location is within or near an area of stressed or unusual vegetation pattern.
- V. Ground water monitoring well is located downgradient from a suspected source area.

#### SAMPLING INTERVAL AND NUMBER OF SAMPLES

- W. The suspect area is small in size (1 to 10 sq ft) and well defined, either because of physical appearance or to evaluate a single-point area. Closely-spaced (2 to 5 ft) lateral composites of from 1 to 4 samples are specified to characterize the single-point area.
- X. The suspect area is large in size (greater than 100 sq ft or longer than 100 ft). Generally, in the larger compositing areas, there are few features to suggest whether or not a problem exists, other than a historic report of past activities in the general area. Samples are spaced from 20 to 50 ft apart and composited. It was generally attempted to limit the number of samples in the composite within the range of 6 to 10, since sensitivity to detecting a hot spot is reduced as the number of samples in the composite increases.
- Z. The sampling interval for vertical samples is self-explanatory on Table S-2. The objective for obtaining depth samples is to determine the vertical distribution of buried materials or to evaluate the limits of vertical migration from a near-surface source material.

*In subsurface stratigraphy  
adequately defined to locate movement  
versus contaminant movement.*

ATTACHMENT 3  
MAGNETOMETER SURVEY PROTOCOL

ATTACHMENT 3  
MAGNETOMETER SURVEY PROTOCOL

A grid system will be established upon the ground surface and at the locus of each point a the total magnetic field will be measured. The grid spacing will be sufficient to detail the site(s) location and boundaries.

A Geometric proton magnetometer, Model G-816/G826 or the equivalent, will be used to conduct the survey. The magnetometer will be operated in accordance with the operating manual.

A base station will be established outside the survey area in an area with no known buried or surface ferrous-metallic objects. Readings at the base station will be repeated every one (1) hour and at the beginning and end of each day. At each point of the grid system, the station location and magnetometer readings will be entered into the geologist's field book for later reduction.

## ATTACHMENT 4

### ELECTROMAGNETIC TERRAIN CONDUCTIVITY SURVEY PROTOCOL

ATTACHMENT 4

ELECTROMAGNETIC TERRAIN CONDUCTIVITY SURVEY PROTOCOL

A grid system will be established upon the ground surface and at the Locus of each point. A measurement of conductivity will be made by the Geologist at each locus of points throughout the grid with supplementary measurements made if deemed necessary.

The instrument utilized will be a GEONICS EM-31 portable, Terrain Conductivity meter. The instrument shall be operated in accordance with Guidelines outlined in the operating manual. A permanent record of results taken in millimhos per meter obtained will be entered into the geologist's filed book for later reduction.

ATTACHMENT 5

ANALYTICAL PROCEDURES FOR EXPLOSIVES IN SOILS



METHOD NO.: 8H

DATE: 4-21-83

EXPLOSIVES IN SOIL BY HPLC

2. APPLICATION: Determination of the following nitro-compounds in soil.

HMX	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
RDX	Hexahydro-1,3,5-trinitro-s-triazine
NB	Nitrobenzene
1,3-DNB	1,3-Dinitrobenzene
1,3,5-TNB	1,3,5-Trinitrobenzene
2,4-DNT	2,4-Dinitrotoluene
2,6-DNT	2,6-Dinitrotoluene
2,4,6-TNT	2,4,6-Trinitrotoluene
Tetryl	2,4,6-Trinitrophenylmethylnitramine

A. Tested Concentration Range:

HMX	0.376-188 ug/g
RDX	0.253-127 ug/g
NB	0.197-98.4 ug/g
1,3-DNB	0.242-121 ug/g
1,3,5-TNB	0.215-107 ug/g
2,4-DNT	0.240-120 ug/g
2,6-DNT	0.217-109 ug/g
2,4,6-TNT	0.301-151 ug/g
Tetryl	0.265-133 ug/g

B. Sensitivity: Peak height near the detection limit. (1 mm = 28 arbitrary units on the integrator readout.) Representative chromatogram near the detection limit can be found in Appendix I.

Peak Height in mm at  
an Attenuation of 2-2

HMX	12 mm for 0.754 ug/g
RDX	18 mm for 0.506 ug/g
NB	11 mm for 0.394 ug/g
1,3-DNB	23 mm for 0.485 ug/g
1,3,5-TNB	20 mm for 0.430 ug/g
2,4-DNT	16 mm for 0.480 ug/g
2,6-DNT	9 mm for 0.434 ug/g
2,4,6-TNT	19 mm for 0.602 ug/g
Tetryl	10 mm for 0.530 ug/g

C. Detection Limits:

HMX	0.376 ug/g
RDX	0.474 ug/g
NB	0.197 ug/g
1,3-DNB	0.242 ug/g
1,3,5-TNB	0.231 ug/g
2,4-DNT	0.240 ug/g
2,6-DNT	0.217 ug/g
2,4,6-TNT	0.301 ug/g
Tetryl	0.265 ug/g

D. Interferences:

1. Any compound that is extracted from soil that gives a retention time similar to the nitro-compounds and absorbs U.V. at 250 nm.
2. Millipore GFWP-01300 filter type GS pore size 0.22 micrometers dissolve in the solvent used.
3. Tetryl and 2-amino-4,6-dinitrotoluene coelute. If a tetryl peak is found in samples, pH adjustment is necessary to separate the peaks to determine which compound is present.
4. 2,4,6-Trinitrobenzaldehyde decomposes rapidly in water solution. Once the acetonitrile standard is made into mobile phase this becomes a problem.

E. Analysis Rate:

After instrument calibration, one analyst can analyze two samples in one hour. One analyst can conduct sample preparation at a rate of three samples per hour. One analyst doing both sample preparation and the HPLC analysis can run 16 samples in an 8-hour day.

II. CHEMISTRY:

A. Chemical Abstracts Service Registry Number:

HMX	2691-41-0
RDX	121-82-4
NB	98-95-3
1,3-DNB	99-65-01
1,3,5-TNB	99-35-4
2,4-DNT	121-14-2
2,6-DNT	606-20-2
2,4,6-TNT	118-96-7
Tetryl	479-45-8

USATHAMA CERT.  
EXPLOSIVES IN SOIL

1. Chemical Reactions:

1. RDX and HMX can undergo alkaline hydrolysis.
2. RDX and HMX degrade at temperatures greater than 80°C in an organic solvent.

2. Physical Properties:

	Formula	Mol. Wt.	M.P. (°C)	B.P. (°C)
HMX	$C_4H_8N_8O_8$	296.16	276	-
RDX	$C_3H_6N_6O_6$	222.12	205	-
NB	$C_6H_5NO_2$	123.11	6	211
1,3-DNB	$C_6H_4N_2O_4$	168.11	90	302
1,3,5-TNB	$C_6H_3N_3O_6$	213.11	122	315
2,4-DNT	$C_7H_6N_2O_4$	182.14	71	300 (decomposes)
2,6-DNT	$C_7H_6N_2O_4$	182.14	66	-
2,4,6-TNT	$C_7H_5N_3O_6$	227.13	82	240 (decomposes)
Tetryl	$C_7H_5N_5O_8$	287.15	131	187

II. APPARATUS:

- A. Instrumentation: Perkin Elmer series 4 High Performance Liquid Chromatograph (HPLC) equipped with a Perkin Elmer ISS-100 Auto-Injector and Perkin Elmer variable wavelength detector LC-75. Hewlett Packard 3390 recording integrator in peak height mode was used to record the data output.

3. Parameters:

1. Column: Two columns are used in series, in the order listed.

a. DuPont Permaphase<sup>R</sup> ODS guard column.

b. DuPont Zorbax<sup>R</sup> ODS 4.6 mm i.d. x 25 cm HPLC  
column with a particle size of 5-6 microns.

2. Mobile Phase: The water/methanol ratio must be adjusted as  
described in the calibration Section V C to obtain optimum  
peak separation.

44-50% water

28-34% methanol

22% acetonitrile

3. Flow: 1.6 mL/min with a pressure of approximately 2860 psig.

4. Detector: 250 nm

5. Injection Volume: 50 uL.

6. Retention Times:      Minutes

HMX	3.38
RDX	4.21
NB	7.33
1,3 DNB	6.63
1,3,5-TNB	5.74
2,4-DNT	9.89
2,6-DNT	9.50
2,4,6-TNT	8.93
Tetryl	7.98

C. Hardware/Glassware:

1. Syringes: 25 uL, 50 uL, 100 uL, 250 uL,  
5 mL gas tight syringe (Hamilton 1005 TEFL)

2. Serum vials with crimp caps and Teflon-lined septa  
Nominal volume of 0.25 mL, 1 mL, 5 mL.

3. Pasteur pipettes and disposable micropipettes.

4. 13 mm stainless steel syringe filter holder  
(Rainin Instrument Co., Inc. #38-101)

C. Hardware/Glassware: (continued)

5. 13 mm x 0.5 micron fluorocarbon filter  
(Rainin Instrument Co., Inc. #38-103 Zefluor disc)
6. Whatman 10 mm glass microfiber prefilter
7. U.S. Sieve series 600 (30 mesh)
8. Aluminum foil pans
9. Liquid chromatograph column 1" o.d. x 12"
10. 2 mL, 3 mL, and 5 mL pipettes

D. Chemicals:

1. Acetonitrile, distilled in glass for HPLC use
2. Methanol, distilled in glass for HPLC use
3. Ethyl Ether, distilled in glass for HPLC use
4. Hexane, distilled in glass for HPLC use
5. ASTM Type II Water
6. SARMS for the nitro-compounds

J. STANDARDS: All concentrations are based on a stock solution concentration of 2000 mg/L. Appropriate adjustments should be made if actual concentration varies from this figure.

A. Calibration Standards:

1. Stock Calibration Standards: Stock solutions containing approximately 2000 mg/L of a nitro-compound are prepared by accurately weighing 10 mg of a SARM into a 5 mL serum bottle and dissolving the nitro-compound in 5 mL of acetonitrile pipetted into the bottle. All compounds appear to be stable for 3 months.
2. Intermediate Calibration Standards: All compounds appear to be stable for 3 months.
  1. Intermediate Calibration Standard A (high level): Add the following volumes of stock calibration standard and seal with a Teflon-lined septum cap. Store in the dark @ 0°-4°C. The resulting solution (5.8 mL) will have the concentrations indicated in the following table.

USATHAMA CERT.  
EXPLOSIVES IN SOIL BY HPLC

A. Calibration Standards: (continued)

Intermediate Calibration Standard A

Nitro-compound	Amt. (uL) of Stock Cal. Std. to add	Resulting conc. (ug/mL)
HMX	1000	345
RDX	600	207
NB	400	138
1,3-DNB	500	172
1,3,5-TNB	500	172
2,4-DNT	500	172
2,6-DNT	500	172
2,4,6-TNT	700	241
Tetryl	600	207
TNBA*	500	172

\*2,4,6-Trinitrobenzaldehyde was originally included for certification. However, the compound is too unstable in water solutions to obtain reproducible certification data. It was included in this table as it affects the total volume used to calculate concentration of the other nitro-compounds.

b. Intermediate Calibration Standard B (low level):

Pipette 4.5 mL of acetonitrile into a 5-mL serum vial. Add 500 uL of Intermediate Calibration Standard A. Seal with a Teflon-lined septum cap and store in the dark @ 0-4°C. The resulting solution (5.0 mL) will have the concentrations indicated in the table below:

Intermediate Calibration Standard B

Nitro-Compound	Resulting conc. (ug/mL)
HMX	34.5
RDX	20.7
NB	13.8
1,3-DNB	17.2
1,3,5-TNB	17.2
2,4-DNT	17.2
2,6-DNT	17.2
2,4,6-TNT	24.1
Tetryl	20.7

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EXPLOSIVES IN SOILS BY RFLC

A. Calibration Standards: (continued)

3. Working Calibration Standards: To a series of ten 5-mL serum vials, approximately one gram of prepared soil (see section V.B.) is accurately weighed into each vial. Using a syringe, the volumes of intermediate standard solutions indicated in the following table are injected onto soil. The serum vial is covered with a septum and shaken until the soil no longer looks wet (approximately 60 seconds). The septum is removed and the indicated amount (see Table below) of acetonitrile is pipetted onto the soil. The septum is replaced and the cap crimped on the vial. The sealed sample is blended on a vortex mixer for approximately 2-3 minutes. The sample is prepared via the procedure given in this method, to give the target concentrations in the following table.

WORKING CALIBRATION STANDARDS

Vial No.	Amt. (uL) Intermed. Cal. Std. to Add		Amt. (mL) (uL) Aceto- Nitrile to Add	Resulting Concentration (ug/g)				NB
	A	B		HMX	2,4,6- TNT	Tetryl	1,3-DNB; 1,3,5-TNB; 2,6-DNT; 2,4-DNT	
1	0	0	2.0	0	0	0	0	0
2	-	12	2.0	0.414	0.289	0.248	0.206	0.166
3	-	24	2.0	0.828	0.578	0.497	0.413	0.331
4	6	-	2.0	2.07	0.145	1.42	1.03	0.828
5	12	-	2.0	4.14	2.89	2.48	2.06	1.66
6	24	-	2.0	8.28	5.78	4.97	4.13	3.31
7	60	-	2.0	20.7	14.5	14.2	10.3	8.28
8	120	-	1.9	41.4	28.9	24.8	20.6	16.6
9	240	-	1.8	82.8	57.8	49.7	41.3	33.1
10	600	-	1.4	207	145	142	103	82.8

- B. Control Spikes: Control spikes are prepared in the same manner as the calibration standards.

C. PROCEDURE:

NOTE THE FOLLOWING SAFETY PRECAUTIONS:

1. A 5-mL gas tight syringe (Hamilton 1005 TEFL) is used, as the teflon/glass seal is less likely to cause an explosion than glass/glass.

USATHAMA CERT.  
EXPLOSIVES IN SOILS

2. The nitro-compounds are less reactive when wet, so every precaution should be taken to ensure that work areas are kept clean and that solutions are not left unattended and allowed to dry.
3. The filtering apparatus is immersed in a water bath and disassembled under water immediately after use. The danger here is solution getting dried on the threads of the filtering apparatus and detonating.
4. When preparing SARM stock standards from pure compounds which are stored in water, small aliquots are scooped onto a nylon or polyvinylidene chloride filter. The water is vacuum filtered off and an appropriate quantity of the "dried" material is weighed out for stock standard preparation. Any extra compound thus dried is disposed of.
5. Prior to working with explosives, it is advisable to discuss safety/handling/storage requirements with an explosives expert.

A. Sample Preparation: The soil sample is removed from the sample bottle and spread out in aluminum foil trays. The sample is air dried. The dried soil is screened through a US series 600 sieve (30 mesh). This screened sample is subsampled according to ASTM procedure D346. The moisture content is determined by ASTM Method D2216-71.

B. Extraction:

1. Accurately weigh 1 gram of prepared soil (see section V.A. above) into a 5-mL serum vial, and pipette 2 mL of acetonitrile onto the soil.

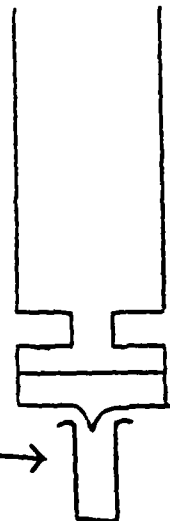
Place a septum and cap on the vial, crimp into place, and shake the vial thoroughly on a vortex mixer for 2-3 minutes.

2. Set up the filtering apparatus, as shown.

5-mL syringe barrel (plunger removed)

5-mL syringe fitted with a Rainin 13 mm stainless steel filter holder with a 10 mm glass microfiber prefilter and a 0.5 micron fluorocarbon filter.

1 mL serum vial to collect filtered sample





f. PROCEDURE: (continued)

3. Prepare the sample for injection as follows:
  - a. Pour the sample extract into the syringe.
  - b. Place the plunger in the syringe and force at least 500 uL of the filtrate into a 1-mL serum vial.
  - c. Using a disposable micropipette, accurately measure 200 uL of filtered extract into a 1-mL serum vial. Accurately measure 600 uL of a 33% methanol/67% water solution onto the filtered sample. This will produce 800 uL of extracted sample in mobile phase.
  - d. Place a septum and cap on the vial and crimp into place. Shake the vial well to thoroughly mix. Store in the dark @ 0-4°C until ready to analyze.
4. For samples outside the calibration range, a smaller sample volume is extracted into 5-mL of acetonitrile.
  - a. Accurately weigh 0.2 gram of prepared soil into a 5-mL serum vial, and pipette 5 mL of acetonitrile onto the soil. Place a septum and cap on the vial, crimp into place, and shake the vial thoroughly on a vortex mixer for 2-3 minutes.
  - b. Prepare the sample for injection as follows:
    - 1) Pour the sample extract into the syringe.
    - 2) Place the plunger in the syringe and force at least 3 mL of the filtrate into a 5-mL serum vial.
    - 3) Using a disposable pipette, accurately measure 1 mL of filtered extract into a 5-mL serum vial. Accurately measure 3 mL of a 33% methanol/67% water solution onto the filtered sample. This will produce 4 mL of extracted sample in mobile phase.

Alternately, the sample extract and methanol/water solution may be accurately weighed into a 5-mL serum vial. (1 mL  $\approx$  1 g)

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- 4) Place a septum and cap on the vial and crimp into place. Shake the vial well to thoroughly mix. Store in the dark @ 0-4°C until ready to analyze.
- c. If the solution prepared from the 0.2 g sample is still above the calibration range, make dilutions of the extract obtained in 4b(1) by taking an appropriate aliquot and adding mobile phase (e.g. 100 mg of acetonitrile sample extract in 20 mL mobile phase) to produce a solution within the calibration range of the instrument.

C. Instrument Calibration/Sample Analysis:

1. Using the auto-injector manufacturer's recommended procedure, introduce 50 uL of the 2X working calibration standard into the chromatographic system. Check the chromatogram to ensure separation of the nitrated toluenes and separation of the nitrobenzene and tetryl. If necessary, adjust the water/methanol ratio of the mobile phase until separate peaks are distinguished. As the column ages, less methanol is required. Generally, the column ages rapidly the first 24 hours, after which it is fairly stable.
2. Once good peak separation is obtained, introduce 50 uL of each working calibration standard and sample into the chromatographic system using the auto-injector manufacturer's recommended procedure.

E. CALCULATIONS:

$$A. \text{ Sample Concentration (ug/g)} = \frac{(\text{peak ht.} - K) \times C \times E}{\text{slope} \times A \times B \times D}$$

where:

- K = y-intercept of the calibration curve regression line
- slope = slope of the calibration curve regression line
- A =  $\frac{8 \text{ mL mobile phase}}{1 \text{ gram sample}}$  = a constant for this method.

Explanation: the instrument reads the total ug in the 50 uL aliquot of sample injected. This constant enables results to be interpreted as ug/g, as the calibration curve in ug/g is obtained by

$$\frac{2 \text{ mL acetonitrile to extract}}{1 \text{ gram calibration std. sample}} \times \frac{4 \text{ mL mobile phase}}{1 \text{ mL acetonitrile extract}}$$

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C. CALCULATIONS: (continued)

- B = sample weight  
C = mL acetonitrile used to extract sample  
D = mL acetonitrile extract diluted into mobile phase  
E = final volume in mL of mobile phase prepared for injection

NOTE: When samples are prepared the same as the calibration standards (1 gram extracted into 8 mL of mobile phase), the above calculation becomes:

$$\begin{array}{l} \text{Sample} \\ \text{Concentration} \\ \text{(ug/g)} \end{array} = \frac{(\text{Peak height} - K)}{\text{slope}}$$

- B. All soils data must be reported on a moisture-free basis. Moisture content is determined by ASTM D2216-71. 100%-% Moisture = % solids.

$$\begin{array}{l} \text{Concentration on a} \\ \text{moisture free basis} \end{array} = \frac{\text{analyte concentration}}{\% \text{ solids}} \times 100$$

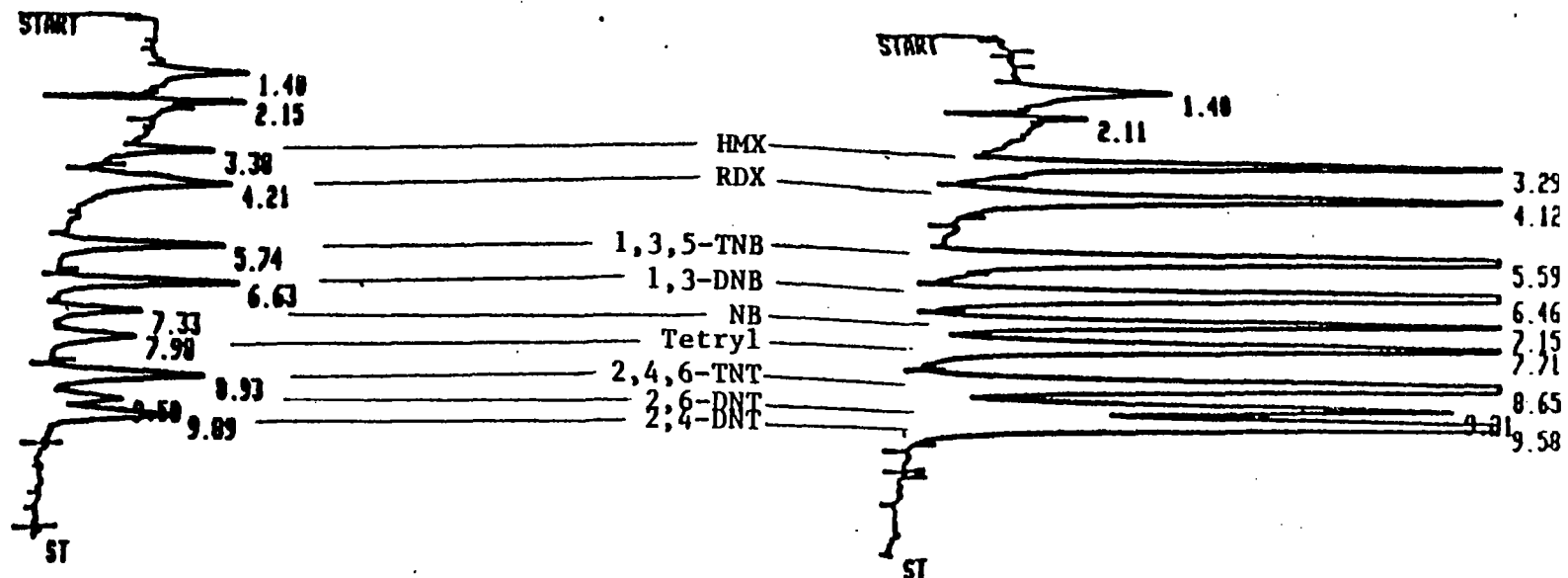
D. REFERENCES:

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B. USATHAMA Method 8H Explosives in Water by HPLC, 12-27-82.

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EXPLOSIVES IN SOILS BY HPLC

APPENDIX I: CHROMATOGRAMS

EXPLOSIVES IN SOIL - ACETONITRILE EXTRACTION



RUN # 145  
ID 1

APR/22/83 12:58:26

HEIGHTX

RT	HEIGHT	TYPE	AR/HT	HEIGHTX
1.40	583	VV	0.435	11.623
2.15	535	PB	0.135	10.666
3.38	332	VB	0.155	6.619
4.21	520	BV	0.365	10.367
5.74	573	PB	0.201	11.423
6.63	632	BP	0.200	12.600
7.33	305	PV	0.208	6.081
7.98	280	VB	0.250	5.582
8.93	539	BV	0.251	10.746
9.50	259	VV	0.243	5.164
9.89	458	VB	0.286	9.131

RUN # 148  
ID 1

APR/22/83 14:04:0

HEIGHTX

RT	HEIGHT	TYPE	AR/HT	HEIGHT
1.40	596	BV	0.344	2.10
2.11	444	PV	0.137	1.57
3.29	2399	PB	0.158	8.48
4.12	2392	BB	0.207	8.45
5.59	4384	PB	0.186	15.58
6.46	4822	PB	0.191	17.05
7.15	2182	BV	0.193	7.71
7.71	2286	VB	0.247	8.00
8.65	3505	BV	0.241	12.39
9.21	1830	VV	0.227	6.00
9.50	3441	VB	0.251	9.13

ATTACHMENT 6

ANALYTICAL PROCEDURE FOR DIOXINS AND DIBENZOFURANS

# ACKNOWLEDGMENT

Thanks J. C. T. Hollander for helpful discus-

No. H<sub>2</sub>SO<sub>4</sub>, 7664-93-9.

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## Determination of Part-per-Trillion Levels of Polychlorinated Benzenofurans and Dioxins in Environmental Samples

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A new method permits determinations of parts-per-trillion levels and lower of tetrachloro through octachloro congeners of dibenzo-*p*-dioxins and dibenzofurans in various environmental tissues and sediments. Preliminary tests indicated the method is applicable to determinations of polychlorinated biphenyls through hexachloro congeners of ortho-unsubstituted biphenyls. Interferences both from natural and from xenobiotic substances are reduced to extremely low levels. The procedure has an extremely low susceptibility to false-positive determinations which could result from the presence of a wide variety of cocontaminants. This approach to contaminant enrichment has permitted reduction of seven processes into a two-step procedure, thereby reducing time requirements and the number of calculations, and making the procedure amenable to automation. The reliability and accuracy of the procedure was demonstrated by the results of intralaboratory and interlaboratory studies and by successful analyses of over 200 samples of a wide variety of types.

Polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), and ortho-unsubstituted polychlorinated biphenyls (non-ortho PCBs) are three chemically and toxicologically related families of anthropogenic compounds that have in recent years been shown to have the potential to cause serious environmental pollution (1-6). These substances are trace-level components in several large-volume and widely used commercial products, principally PCBs and chlorinated phenols, and can also be produced during combustion processes (7,8) and by photolysis (12, 13). In general, PCDDs, PCDFs, and non-ortho PCBs are classified as highly toxic compounds (4), although the toxicities are dramatically de-

pendent on the number and positions of the chlorine substituents (15). About 10 individual members of a total of 216 PCDDs, PCDFs, and non-ortho PCBs are among the most toxic man-made or natural substances to a variety of animal species (1-4). The toxic hazards posed by these chemicals are exacerbated by their propensity to persist in the environment (16-18) and to readily bioaccumulate (19-21), and although the rate of metabolism and elimination is strongly species dependent (20), certain highly toxic isomers have been observed to persist in the human body for more than 10 years (22).

The majority of scientific and governmental concerns for the hazards of these compounds have been directed toward analytical methodologies, toxicology, epidemiology, and determination of the disposition in the environment of the single most toxic isomer, 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD) (1-6, 8).

More recently, however, investigations into the formation and occurrence of PCDFs suggest that this family of toxic compounds may commonly occur at comparable or greater levels than and could generally pose a greater hazard than PCDDs. PCDFs are often found as cocontaminants in and are readily produced from pyrolysis of PCBs (7, 23-26). Most important, the PCDFs produced from pyrolysis of PCBs are predominantly the most toxic isomers, those having a 2,3,7,8-chlorine substitution pattern (5). A number of recent fires involving electrical transformers and capacitors have demonstrated the potential for formation of hazardous levels of PCDFs from pyrolysis of PCBs (26-28, 30).

In light of these findings and because of the dearth of data pertaining to the occurrence of these compounds in the environment, PCDFs and non-ortho PCBs were included as target compounds in a proposed survey by this laboratory of important U.S. rivers and lakes for PCDDs. The decision to include as many PCDD isomers as possible was based on

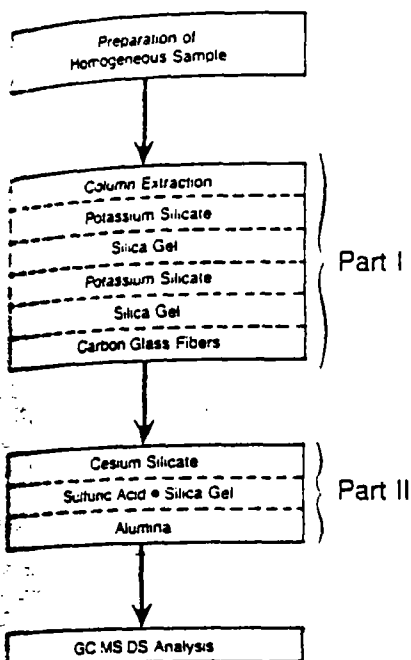


Figure 1. Flow chart of total procedure.

(1) several other PCDD isomers are also ex-  
 toxic (15); (2) pentachlorophenol, a large-volume  
 and wood preservative, contains relatively high levels  
 hepta-, and octachlorodibenzodioxins and essentially  
 CDDs (7, 8, 29); and (3) incineration of materials con-  
 chlorophenols readily produces mixtures of PCDDs,  
 TCDD is a minor component. On the other hand,  
 toxic 1,2,3,7,8-pentachloro isomer is a major com-  
 PCDD incineration products of pentachlorophenol  
 Component-specific analyses can be a crucial link to  
 of contamination because different sources of  
 and PCDFs usually produce mixtures of distinctly  
 relative component abundances (7). On the other  
 the preferential accumulation of certain isomers in  
 may prevent source identification from analyses of  
 equal samples.

Analytical method developed for this investigation was  
 to meet six criteria: (1) permit determinations of  
 of these compounds, especially those possessing  
 three chlorine substituents; (2) permit isomer-  
 determinations of the most toxic or otherwise im-  
 components; (3) provide a lower limit of detection for  
 components of between 1 and 5 parts per trillion  
 a variety of environmental samples; (4) generate data  
 acceptable and adequately defined level of accuracy  
 precision; (5) exhibit a very low and well-defined sus-  
 to interference and false-positive determinations;  
 minimize analyst's time requirements to permit  
 of large numbers of samples.

Determinations of PCDDs and PCDFs demand an unusu-  
 level of analytical assurance, not only because of the  
 hazards of these compounds, the intensity of public  
 concern, and the widespread nature of the  
 but also because of the great difficulties in rigorous  
 of individual isomers. These difficulties are not  
 related to the problems of distinguishing between  
 this problem is essentially solved (31-34)—but are  
 to the possibility of specific and nonspecific inter-  
 from natural and especially xenobiotic substances

herein are the description of an analytical method  
 of validation and applications studies which  
 accuracy and reliability and demonstrate the utility

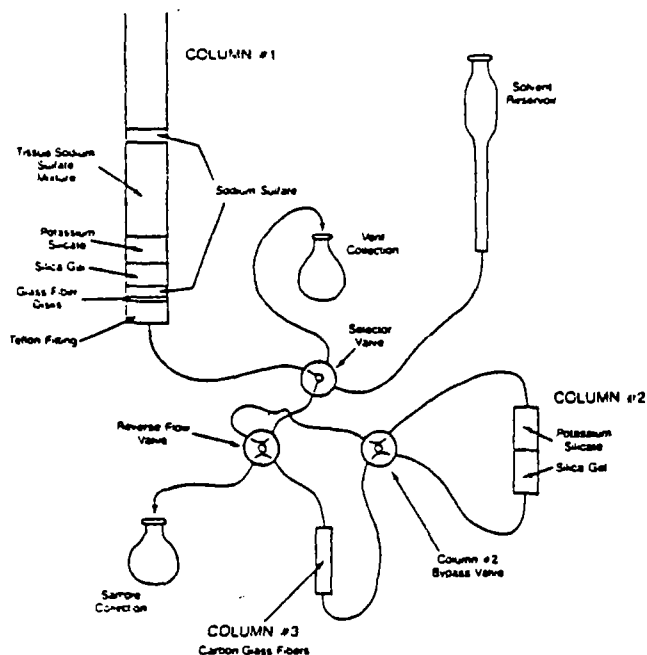


Figure 2. Schematic of part I enrichment apparatus.

of the method developed for the determination of PCDDs,  
 PCDFs, and non-ortho PCBs in a variety of environmental  
 matrices.

## EXPERIMENTAL SECTION

**Enrichment Procedure.** Tissue and sediment or soil samples (spiked with isotopic marker compounds) are processed in a two-part enrichment procedure (Figure 1). In part I, a mixture of the sample and sodium sulfate is subjected to solvent extraction, and the extract is, in the same process, passed through a series of silica-based adsorbents and then through the carbon/glass fiber adsorbent. The extract passes through the adsorbents in the following order: potassium silicate, silica gel, cesium or potassium silicate, silica gel, and finally an activated-carbon adsorbent. The residues of interest [PCDDs, PCDFs, and non-ortho PCBs, as well as other chemical classes such as polychlorinated naphthalenes (PCNs), polychlorinated biphenylenes, and certain polynuclear aromatic hydrocarbons] are retained on the carbon adsorbent and subsequently recovered by reverse elution with toluene.

In part II of the procedure, following a change of solvent to hexane, the sample is applied to a second series of adsorbents contained in two tandem columns. The first column contains small amounts of cesium or potassium silicate and sulfuric acid impregnated silica gel. The effluent from this column flows directly onto the second column containing activated alumina on which the final fractionation of several classes of residues is accomplished. Following reduction of sample volume, analyses are carried out by high-resolution gas chromatography/low-resolution mass spectrometry/computer data system analysis (HRGC/LRMS/DS) and under some circumstances by gas chromatography/electron capture detector analysis (GC/EC).

**Part I.** The components of the apparatus used in part I of the enrichment procedure are depicted in Figure 2. Column 1 (about 4.5 cm i.d. and about 1 m long) is connected to column 2 (22 mm i.d. × 100 mm, Michel-Miller precolumn 5769-34, Ace Glass, Vineland, NJ) and to column 3 (1.0 cm i.d. × 6 cm thick-walled, precision-bore glass tubing, Kontes, Vineland, NJ) by means of standard 1/16 or 1/8 in. o.d. Teflon tubing and tube end fittings. Column 3 is equipped with in-house fabricated Teflon fittings. The solvent flow switching valves are Hamilton miniature inert valves (Hamilton Co., Reno, NV): selector valve (no. 86781), on-off valve (no. 86775), and bypass and reverse-flow valves (no. 86781). The washing solvent reservoir is constructed of a 20-cm length of 12 mm o.d. glass tubing and a 200-mL reservoir fitted with a 24/40 female ground glass joint. The valving arrangement (Figure 2) is designed to enable the analyst to perform the following operations: venting of the solvent line from column 1, venting of the solvent reservoir, bypass of column 2, delivery of



... from column 1 to columns 2 and 3 sequentially, solvent from the reservoir sequentially to columns 2 and 3 only, reversal of solvent flow in columns 2 and 3 only, reversal of solvent flow in all lines. The solvent is continuously pressurized with 1-10 psi nitrogen during the process. Column 2 is packed with equal volumes, 15 cm each, of cesium or potassium silicate and silica gel (EM-60, 60 mesh) bracketed by plugs of glass wool or preferably fiber filters (3- $\mu$ m retention GF/D, Whatman Inc., Clifton, NJ). Column 3 is packed with a mixture of Amoco PX-21 carbon and glass fibers as described previously (36). The column is packed in the following sequence: two disks of glass fiber (GF/D, 4.7-cm diameter, Whatman Inc., Clifton, NJ), 30 g of anhydrous sodium sulfate, 30 g of silica gel (130  $^{\circ}$ C activated), 30 g of potassium silicate (130  $^{\circ}$ C activated), 50 g of a 1:4 (w/w) mixture of the sample and anhydrous sodium sulfate, and lastly a 2-cm depth of anhydrous sodium sulfate.

Column 4 (Figure 2) is usually packed with fresh adsorbents but this column can be used for more than one sample. The amounts of extracted materials, such as lipids, are determined. The carbon adsorbent in column 3 is routinely reused (under 3-8 psi of nitrogen) between samples by the following solvent sequence: 100 mL each in reverse flow of methanol, toluene, and cyclohexane/methylene chloride (solvent A). Column 2 is bypassed during these washings. At the final washing with solvent A, which is directed through column 2 in the reverse direction to remove residual air contaminants. Care must be taken to avoid passing solvent through column 2. Another 100 mL of solvent A is passed through columns 2 and 3 in the forward direction to complete the solvent washing. Complete displacement of toluene from column 3 is essential. After columns 2 and 3 are properly washed, column 1 is loaded with adsorbents and sample, a small amount (usually 100  $\mu$ L) of marker compounds is applied to the column and washed onto the packings with four or five 20-mL portions of solvent A using a Teflon wash bottle. The selector valve is positioned so that column 1 is connected to the flask and air is allowed to escape. The flow of air from the flask is monitored as it bubbles through solvent at the vent. After the sample is spiked with marker compounds, solvent A is carefully applied to column 1, and the position of the solvent front is observed. As the solvent front reaches the transfer line (about 1 m in length), air bubbles in the line are removed by stopping the flow and tapping the line. When the solvent front reaches the selector valve, the valve is repositioned to extract through columns 2 and 3, and the enrichment procedure is under way. The effluent is collected in a flask positioned above columns 2 and 3 to maintain a positive pressure on these columns. The height of column 1 above the collection flask is adjusted to produce a solvent flow of not less than 3 mL/min but sufficient to complete the process. Occasionally the solvent flow will slow or stop during the process and will require the application of 1 or 2 psi of nitrogen to the system at column 1. Rarely, the glass fiber at the inlet end of column 3 become clogged during the process of decomposition or very oily (especially lake trout) samples. To reduce these complications, a removable column (1.0 m  $\times$  3 cm) containing 4 or 5 disks of glass microfibers is inserted in line at the exit end of column 2. If this filter becomes clogged, it can be replaced during the process.

After completion of the initial extraction/adsorption operation, column 3 (bypassing column 2) is washed in the forward direction with 75 mL of solvent A and then 50 mL of methylene chloride/benzene (75/20/5) at a flow of approximately 2 mL/min. These washings are collected in the flask with the sample. The reservoir is then charged with 40 mL of toluene and passed through the carbon (column 3) in the reverse flow at approximately 2 mL/min and collected in a 100-mL flask (24/40). At this point, part I of the procedure is complete.

The sample in toluene is subjected to rotary evaporation at a vacuum of about 0.1-0.2 atm. The rotary evaporator must be maintained in an uncontaminated condition and washed with organic solvents. No lubricating greases are used. The integrity of the sample is protected during rotary

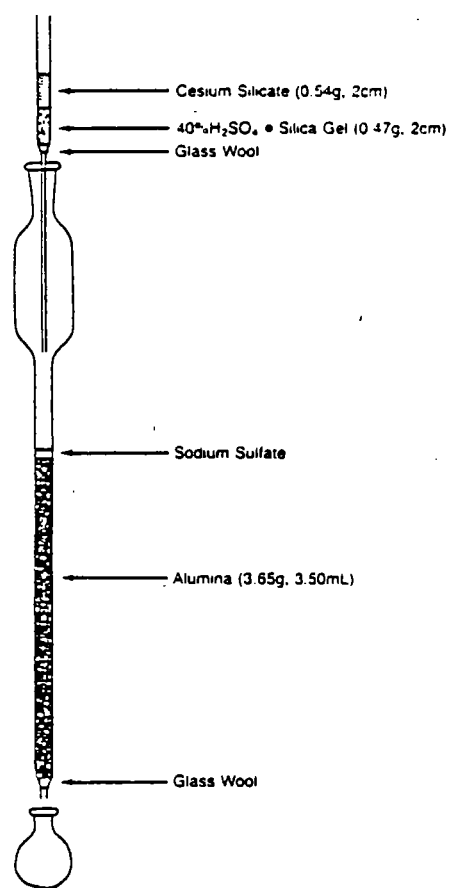


Figure 3. Schematic of part II enrichment procedure.

evaporation by the use of a vapor trap situated between the sample flask and the evaporation apparatus; the vapor trap is thoroughly washed with toluene between samples. The toluene solution (sample) is carefully reduced to less than 1 mL or just to dryness and removed immediately. The solution or dry sample can be stored in a freezer. At this point, the sample is ready for part II of the procedure (after removal of all toluene).

**Part II.** The apparatus for part II of the enrichment procedure consists of two columns arranged in tandem (Figure 3). Column 4 is prepared from a disposable Pasteur pipet and is packed first with a plug of glass wool, then with 3 cm (0.50 g) of sulfuric acid impregnated silica gel, then with 3 cm (0.54 g) of cesium potassium silicate (not heat activated), and finally with 0.5 cm of anhydrous sodium sulfate. Column 5 is constructed from a 5-mL graduated pipet fitted with a 20-mL reservoir and a ground-glass joint. Column 5 is packed with a plug of glass wool followed by 3.50 mL (3.65 g) activated (190  $^{\circ}$ C) alumina and then 0.5 cm of anhydrous sodium sulfate. The alumina is packed firmly by sharply tapping the supporting clamp.

Columns 4 and 5 (Figure 3) are thoroughly washed before use, column 4 with 10 mL of hexane and column 5 under approximately 5 psi of nitrogen pressure, with 30-50 mL of hexane to remove entrapped air. Following the washings, column 4 is partly inserted into column 5 so that the effluent from column 4 flows directly onto the adsorbent bed of column 5. A 50-mL collection vessel is placed at the exit end of column 5. Pasteur pipets previously heated for several hours at 500  $^{\circ}$ C are used for liquid transfers. The sample is applied to column 4 by using four to six separate 1-mL washings (approximate volumes) of hexane totaling 5.0 mL. Each washing is allowed to pass through column 4 and completely onto the alumina of column 5 before the next washing is applied. After 5.0 mL of hexane has passed through column 4, this column is discarded, and a second 5.0-mL volume of hexane is then applied to column 5. The following sequence of eluting solvents is then applied to column 5: 15 mL of 2%, then 15 mL of 5%, and finally 20 mL of 8% methylene chloride in hexane. A total of 60 mL of effluent is thus collected in two fractions, the first measuring 23 mL and the second 37 mL. Due to variations in the activities of different lots of alumina, the

sample volumes must be carefully determined for each

fraction, containing the residues of interest, is reduced to a volume to about 0.5 mL under a stream of nitrogen in a 40 °C water bath. The sample is transferred to a conical flask and four 0.5-mL washings with methylene chloride, each being reduced to a smaller volume under a stream of nitrogen before the next is added. Following the last transfer, the solvent is completely evaporated and the appropriate volume of solvent (usually 10  $\mu$ L of toluene or *o*-xylene) is added for analysis. If the analysis is to be performed later, the sample can be kept in the dry state and stored in a freezer. Before analysis, the solution is drawn up into the microliter syringe and applied repeatedly to the wall of conical portion of the flask to bring the entire sample into solution. Gas chromatographic and spectrometric analyses are carried out by the injection technique (no splitting of the sample) with 2–4  $\mu$ L of sample or by the on-column technique in which 1–2  $\mu$ L of sample are injected.

**Sample Preparation.** Tissue and sediment samples are dried with at least 4 times their weight of anhydrous sulfate. Samples are first cut into small pieces, ground in a meat grinder (if necessary), and mixed thoroughly with anhydrous sulfate with a spoon in a glass or polyethylene dish. The mixture is then spread out to a depth of less than 3 cm so that the sample, which solidifies after 3–6 h, can be readily broken up by being overnight. The mixture is then dry-blended (any type of model blender) in a glass jar to yield a fine powder. Samples of low water content did not require overnight equilibration with sodium sulfate before blending. A second blending was required 4–6 h after the first is often required to produce a homogeneous and finely divided sample.

**Instrumental Instruments and Conditions.** Determinations of PCDFs and PCDDs were carried out with a Finnigan 4023 gas chromatograph equipped with an INCOS data system and with electron and positive chemical ionization options. Methane was used as the reagent gas for the negative ion chemical ionization. The gas chromatograph was usually fitted with either a 0.25 mm DB-5 fused-silica capillary column (J&W Scientific Inc., Rancho Cordova, CA) or a 55 m  $\times$  0.27 mm Silar 1000 prepared by H. R. Buser, Swiss Federal Research Institute, Dübendorf, Switzerland. The carrier gas was helium and the following temperature program was routinely used with *o*-tolidine: 150–255 °C at 3 °C/min and then 12 °C/min to 300 °C and hold for 10 min. The electron impact mode (EI) and mass ion detection (MID) were routinely used for GC/MS detection and quantitation of PCDFs and PCDDs including marker compounds ( $[^{13}\text{C}]$ -TCDD,  $[^{37}\text{Cl}]$ -TCDF, and  $[^{37}\text{Cl}]$ -TCDD). By use of DB-5 column, a series of either 8 or 12 scans at  $m/z$  values were monitored within each chromatographic window, each window being defined by lower and upper elution limits of a particular group of PCDF and PCDD congeners. The MID analysis usually involved monitoring of four or five members of a molecular ion cluster resulting from the fragmentation of the fragment ion cluster resulting from the precursor ion,  $M - 63$ .

Chromatographic analyses employing a packed column [2 mm  $\times$  1% OV-17 on 100/120 Supelcoport (Supelco, Inc., Bellefonte, PA)] were carried out on a Varian 3700 gas chromatograph equipped with an electron capture detector. Nitrogen was used as the carrier gas with the following temperature program: 30–270 °C at 8 °C/min and hold for 15 min.

**Materials.** All solvents were glass distilled grades (MC/B, Fisher, OH, or Burdick and Jackson, Muskegon, MI). Silica gel (60–230 mesh (EM Reagent, MC/B, Cincinnati, OH) and AG4, Bio Rad Labs, Richmond, CA) were used. The silica gel was washed with methanol and then methylene chloride and activated at 190 °C for at least 2 days. Silica gel was washed in the same manner and activated at 130 °C. The silica gel (60–230 mesh (MC/B, no. SX760) is heated at 500 °C overnight and stored in screw capped bottles.

PX-21 activated carbon was obtained from the Amoco Chemical Center, Naperville, IL 60566, and lot numbers 75-8, 76-9, and 78-10 were successfully used in this laboratory. The carbon is now commercially available from Anderson De-

velopment Co., Adrian, MI 49221, under the name AX-21.

Potassium and cesium silicates were prepared from the reaction of the corresponding alkali metal hydroxides with silica gel in methanol at 55 °C for 90 min. The reaction is carried out in a 1- or 2-L round-bottom flask which is rotated and heated with a rotary evaporation apparatus (no vacuum applied). Sixty grams of CsOH (99+%, Aldrich Chemical Co., Milwaukee, WI) is dissolved in 200 mL of methanol and separated from insoluble material by decantation. An additional 200 mL of methanol is added followed by 100 g of silica gel. For potassium silicate, 168 g of KOH (J. T. Baker Chemical Co., Phillipsburg, NJ), 300 g of silica gel (EM60), and approximately 700 mL of methanol are used; decantation is not necessary for KOH. Following the reaction, the mixture is poured into a large glass column containing a plug of glass wool. Special care must be exercised to avoid contact with the extremely caustic solution, especially eye contact. The adsorbent is washed into the column with methanol, and then 200 mL of methanol for every 100 g of silica gel is added to the column. The methanol can be pushed through the column under slight gas pressure, and as the level of the liquid reaches the bed of adsorbent, 200 mL of methylene chloride for every 100 g of silica gel is applied. The liquid is pushed through the column and the silicate partly or completely dried under a slow flow of nitrogen. Cesium silicate is dried completely under a stream of nitrogen and is not heat activated; potassium silicate is activated at 130 °C.

Sulfuric acid impregnated silica gel (40% w/w), abbreviated as SA-SG, is prepared by adding 2 parts of concentrated sulfuric acid to 3 parts by weight of 130 °C activated silica gel in a screw capped bottle and shaking until the mixture is completely free of lumps, about 15 min. The silica gel is activated at 130 °C; unactivated silica gel is unsatisfactory for the preparation of SA-SG. The adsorbent is stored in a screw capped bottle.

Nitrogen gas used for evaporations of solvents is passed through a copper tube (40 mm o.d.  $\times$  60 cm) containing activated carbon (coconut charcoal, Fisher Scientific Co., Pittsburgh, PA) bracketed by glass wool and glass microfiber filters. Following the carbon trap, a microfiber filter (Microfibre filter 9802-AAQ, 505-AAQ, 0.3- $\mu$ m retention, Balston Filter Products, Lexington, MA) is inserted in the line in an attempt to prevent movement of carbon particles through the nitrogen line.

## RESULTS AND DISCUSSION

**Development and Functions of the Components of the Enrichment Procedure. Part I.** A primary objective in the initial approach to the development of this method was to make optimum use of the highly selective absorptivity of activated carbons for polychlorinated polycyclic aromatic compounds (37). The carbon adsorbent selected for this procedure was Amoco PX-21 dispersed in glass fibers (CGF) which has been thoroughly evaluated in this laboratory with regard to its selectivity for a wide variety of chemical classes (36, 37). At least four lots of PX-21 carbon have been successfully employed by this and other laboratories (26, 38–46) in analyses of PCDDs and PCDFs.

Application of extracts of whole fish directly to the carbon adsorbent dispersed in glass fibers was found to be generally unacceptable due to the adsorption of biogenic substances causing high back pressures. Pretreatment of the tissue extract with the strongly basic adsorbent potassium silicate (KS) (47, 48) followed by activated silica gel (SG) greatly facilitated the flow of the tissue extract through the carbon adsorbent. Other combinations with alumina and with Florisil or with potassium silicate alone were less effective. The combination of KS, SG, and PX-21 carbon adsorbents achieved a very high degree of enrichment of PCDDs, PCDFs, and non-ortho PCBs. Tissue samples up to 50 g and containing 10–20 g of fat routinely give only submilligram residues in the sample recovered by reverse elution of the carbon with toluene. Integration of these three steps yielded a procedure that permitted simultaneous sample extraction, removal of acidic and highly polar coextractables, and selective adsorption of the compounds of interest onto carbon (part II) and was readily

arrangement which simplified sample, solvent manipulations (Figure 2). Several sets of each be loaded with a sample, the three solvent, and the enrichment processes allowed extended, by gravity solvent flow. The use of combination of potassium silicate and silica gel ensures that the interfering lipid materials from reaching the carbon and permits the accurate estimate the amount of colored lipid material adsorbed by the potassium silicate/silica gel in those cases in which little or no accumulation is observed on column 2, consideration can be made of column 2 for another sample. Cesium silicate compounds more effectively than KS used in column 2 but is 50 times more costly. The operations of part I eliminate the need for which require extensive sample manipulations and labor intensive. Such procedures which are employed in other methods include one or more (1) acidic or basic digestion of the sample, liquid-liquid partitioning steps, (3) Soxhlet extraction, and gel permeation chromatography. The ability of enrichment procedures in a one-step, continuous can result in enhanced recovery and precision to reduced analysis time. Furthermore, this method itself to the possibility of development into a multisample procedure (49).

Chromatography (GPC) was initially employed as an enrichment step preceding the adsorbent but often did not have the capacity for the sample required in these analyses. Furthermore, the use of GPC into the initial enrichment procedure required additional sample extraction and solvent volume steps precede the GPC procedure.

To protecting the adsorptive capacity of the adsorbent, the silicate adsorbent has been demonstrated necessary to remove acidic compounds which represent serious interferences to determinations of PCDDs. The silicate adsorbents retain substances which have acidity constants of 10 and lower, including carboxylic acid compounds and sulfonamides. Aromatic, hydroxy PCBs and hydroxydiphenyl compounds which can produce false-positive GC/MS results are effectively removed by the silicates (35).

Under the conditions of this enrichment procedure, the adsorbent will retain only a limited number of classes of compounds (50), including polyhalogenated planar aromatic compounds, to some extent PAHs with three rings, and strongly acidic compounds that are sequestered by the silicate adsorbent before reaching the carbon. The large majority of synthetic organic compounds which are commonly encountered as persistent contaminants are weakly adsorbed and readily desorb from the carbon by the extraction solvent. Included among the chemicals are compounds which interfere in the determinations of PCDDs, PCDFs, and non-ortho PCBs, DDE, PCBs, methoxy PCBs, polychlorinated biphenyls (PCBPEs), and methoxy PCBPEs (35). The adsorbent also exhibits a very low affinity for the compounds which are not retained by the potassium silicate/silica gel combination.

In part II of the enrichment procedure (Figure 3) the sample is first passed through a strongly basic adsorbent, alumina, and a strongly acidic adsorbent, 40% sulfuric acid impregnated silica gel (SA-SG), in the nonpolar solvent, hexane. The sample is then subjected to chromatography on acid alumina. The use of the sample to cesium silicate in the nonpolar solvent hexane virtually assures the removal of

trace residues of acidic compounds. Use of cesium silicate which has been activated at 130 °C resulted in poor recoveries of hepta- and octachloro isomers. The adsorbent should simply be purged of solvent under a stream of nitrogen after preparation and not oven activated.

The sulfuric acid impregnated silica gel (40% w/w) has been demonstrated in this laboratory and elsewhere (51) to strongly retain or undergo chemical reactions with a number of classes of compounds. A series of polynuclear aromatic hydrocarbons (PAHs) possessing two to four condensed rings was found in this laboratory to be effectively retained by this adsorbent. The adsorbent is also undoubtedly very effective in removing numerous types of compounds by reactions of dehydration, acid-catalyzed condensations, and oxidation as demonstrated by the complete charring and polymerization of tissue extracts applied to this material. Colored bands of adsorbed materials are normally observed on the SA-SG adsorbent following sample application in part II of this procedure. The reactivity of this adsorbent toward PAHs is complementary to the activated-carbon adsorbent which strongly adsorbs certain PAHs which are subsequently recovered with the PCDDs, PCDFs, and non-ortho PCBs. Because polynuclear aromatic hydrocarbons will elute from alumina under the solvent conditions employed in the subsequent step involving alumina chromatography, it is important that PAHs be removed prior to this step. In some environmental samples, especially sediments, high concentrations of PAHs were frequently encountered.

The final step of the enrichment procedure, alumina chromatography, is designed primarily to separate PCDDs, PCDFs, and non-ortho PCBs from polychlorinated naphthalenes (PCNs), trace residuals of PCB isomers, and other polychlorinated aromatic compounds. In addition to PCDDs, PCDFs, and non-ortho PCBs the only classes of compounds which have been shown in this laboratory and elsewhere (46) to be recovered from the carbon are PCNs, polychlorinated biphenyls, and certain polychlorinated PAHs. The alumina chromatography removes the large majority of the 75 possible PCN isomers, but four to six penta- and hexachloronaphthalenes are partially recovered with the PCDDs, PCDFs, and non-ortho PCBs. Use of basic alumina (190 °C activated) requires higher concentrations of methylene chloride to recover PCDDs and PCDFs.

**In-House and Extralaboratory Evaluations and Validation Studies.** The following studies and evaluations were made: (a) determinations of the mean recoveries of a series of representative compounds of the three chemical groups at selected concentrations, (b) determinations of the coefficient of variation associated with each set of recovery data, (c) estimation of the lower limit of detection and determination of the various congener groups or individual components in a variety of sample types, (d) evaluation of the degrees of interference posed by seven series of polychlorinated aromatic compounds which represent the greatest threat of producing false-positive data, and (e) determination of the success rate for completed analyses of approximately 200 environmental samples.

**Recovery Studies.** Initial recovery studies were performed by using an abbreviated procedure which did not incorporate either the silica gel in part I or the alumina chromatography in part II. This procedure was highly effective for the determination of PCDDs, PCDFs, and non-ortho PCBs in biological materials. The major disadvantage of this abbreviated procedure appeared to be the inclusion of a large number of polychlorinated PAHs such as PCNs in the analyte. Nevertheless, an abbreviated procedure excluding alumina chromatography has been successfully used in the analyses of over 30 environmental samples. PCNs were the most significant cocontaminant observed but did not interfere in the deter-

# Recoveries of Selected PCDDs and PCDFs in Salmon Oil from Abbreviated Procedure: Potassium Silicate, Fibers, Cesium Silicate, and Sulfuric Acid-Silica Gel<sup>a</sup>

recoveries of selected compounds						
2,3,6,8-Cl <sub>4</sub> -furan	2,3,7,8-Cl <sub>4</sub> -dioxin	1,2,4,7,8-Cl <sub>5</sub> -furan	1,2,3,4,7,8-Cl <sub>6</sub> -furan	1,2,3,4,6,8,9-Cl <sub>7</sub> -furan	OCDD	OCDF
109 [1]	115 [1]	115 [1]	113 [1]	117 [1]	86 [1]	79 [1]
recoveries of selected compounds						
2,3,7,8-Cl <sub>4</sub> -furan 2,3,7,8-Cl <sub>4</sub> -dioxin <sup>b</sup>	1,2,4,7,8-Cl <sub>5</sub> -furan	1,2,4,6,7,9-Cl <sub>6</sub> -furan	1,2,3,4,7,8-Cl <sub>6</sub> -dioxin	1,2,3,4,6,8,9-Cl <sub>7</sub> -furan	OCDD	OCDF
81 (9) [4]	70 (5) [4]	75 (5) [4]	82 (3) [4]	77 (5) [4]	87 (7) [4]	75 (5) [4]
102 (2) [4]	97 (3) [4]	84 (4) [4]	98 (2) [4]	87 (6) [4]	76 (3) [4]	74 (5) [4]
66 (2) [3]	80 (-) [2]	68 (3) [3]	76 (-) [2]	72 (8) [3]	66 (3) [3]	62 (14) [3]

were determined on a 12-m OV-17 WCOT glass column and electron capture detection (<sup>63</sup>Ni) using helium at 50 cm/s and the temperature program: 190 °C for 2 min, then 4 °C/min to 240 °C and hold 15 min. Numbers in parentheses are coefficients of variation. Numbers in brackets are the number of replicate samples analyzed. <sup>b</sup> 2,3,7,8-TCDD and 2,3,7,8-TCDF coeluted on the OV-17

## Recoveries of Selected PCDDs and PCDFs from Spiked Samples of Homogenized Whole Fish Using the Enrichment Procedure

recoveries of selected compounds								
sample	2,3,6,8- Cl <sub>4</sub> -PCDF	2,3,7,8- Cl <sub>4</sub> -PCDF and PCDD	1,2,4,7,8- Cl <sub>5</sub> -PCDF	1,2,4,6,7,9- Cl <sub>6</sub> -PCDF	1,2,3,4,7,8- Cl <sub>6</sub> -PCDD	1,2,3,4,6,7,9- Cl <sub>7</sub> -PCDF	OCDD	OCDF
spiked carp and spiked PCDD at PCDF (100 ppb)	81 (1) [4]	92 (3) [4]	94 (3) [4]	98 (6) [4]	104 (4) [4]	95 (8) [4]	99 (22) [4]	91 (16) [4]

recoveries of selected compounds				
sample	[ <sup>13</sup> C]-2,3,7,8-TCDD	[ <sup>37</sup> Cl]-2,3,7,8-TCDF	[ <sup>37</sup> Cl]-1,2,7,8-TCDF	[ <sup>37</sup> Cl]-OCDD
samples spiked at 25-50 ppb	82 ± 27 [49]	58 ± 16 [11]	75 ± 18 [10]	83 ± 30 [18]

recoveries of selected compounds <sup>a</sup>									
	Cl <sub>4</sub> PCDFs	Cl <sub>5</sub> PCDFs	Cl <sub>6</sub> PCDFs	Cl <sub>7</sub> PCDFs	OCDF	Cl <sub>3</sub> PCDD	Cl <sub>4</sub> PCDD	Cl <sub>7</sub> PCDD	Cl <sub>8</sub> biphenylene
spiked at 20 ppb	58 ± 10	64 ± 6	64 ± 7	63 ± 10	59	41	49	58	52
spiked at 100 ppb	52 ± 7	55 ± 4	53 ± 6	56 ± 4	52	84	60	51	59

of PCDDs and PCDFs. The recoveries of a series of PCDDs and PCDFs from spiked samples of salmon oil by the abbreviated procedures are given in Table I. Recoveries of spiked fish samples containing up to 20 g of oil were carried out by GC/EC and showed very low levels of matrix components in the analytes (49). Incorporation of silica gel in part I and alumina in part II of the procedure, recoveries of a series of PCDDs and PCDFs from spiked whole fish samples were again determined (Table II). Recently, an independent evaluation of the enrichment procedure was carried out at the University of Guelph and included the determinations of recoveries of spiked fish of a mixture of fourteen tetra-, five penta-, three hepta-, and one octachlorodibenzo-p-dioxin, one hexa-, and one heptachlorodibenzo-p-dioxin and one tetrachlorobiphenylene (45). Mean and standard deviations of the recoveries are presented herein to support the effectiveness of the method for the con-

Only two sets of recovery determinations have been made for three representative non-ortho PCBs spiked at 100 ppb: 3,4,3',4'-tetrachloro (38 and 57%), 3,4,5,3',4'-pentachloro (43 and 47%), and 3,4,5,3',4',5'-hexachloro (54 and 59%).

The demonstration of the effectiveness of recovery of a large selection of PCDD and PCDF isomers, in particular those tetra-, penta-, and hexachloro isomers possessing the critical 2,3,7,8-chlorine substitution pattern, is especially important to defining the comprehensiveness and applicability of the method. The recoveries of all the isomers studied are generally comparable and no particular isomer or group of isomers appear to be selectively excluded by the enrichment procedure.

In addition to the recovery data derived from spiked samples as part of the validation studies, a substantial collection of recovery data was also generated for the four major components of the marker compounds which were added to each sample prior to the enrichment process. The marker compounds, [UL-<sup>13</sup>C]-2,3,7,8-TCDD, [UL-<sup>37</sup>Cl]-OCDD, and a mixture of six [UL-<sup>37</sup>Cl]-TCDFs including [<sup>37</sup>Cl]-1,2,7,8- and [<sup>37</sup>Cl]-2,3,7,8-TCDFs as the major components, were routinely incorporated into each sample at levels of 50, 50, 25, and 25 ppb, respectively. Although the range of recovery data values

Relative Recoveries of Tetrachlorodibenzo-*p*-dioxins from the Unabbreviated Enrichment Procedure\*

MS isomer	TCDD isomer	rel recovery	GC/MS peak no.	TCDD isomer	rel recovery
1368		1.20	8	1234, 1237, 1238, 1246, 1249	1.45
1379		1.27	9	1236, 1279	1.47
1378		1.57	10	1278, 1469	1.35
1369, 1247, 1248		1.47	11	1239	1.39
1268		2.13	12	1269	1.39
1478		1.30	13	1267	2.85
2378		1.00	14	1289	3.62

\* Approximately 2 ng of TCDDs was applied i.d. the enrichment procedure. Determination was made on a 60 m × 0.25 mm i.d. Hewlett-Packard (Inc.) capillary column under MID-EI mass spectrometric conditions: temperature, 200 °C for 1 min, then to 250 °C at 5 °C/min; He carrier gas.

The marker compounds generally reflects the reduced GC/MS/DS quantitation of trace analytes using standard technique, the determinations of the marker compounds in these samples performed nearly a 3-year period provide a practical measure of the enrichment procedure and the analytical method (Table II). The average recoveries of the marker compounds over this extended period were consistently satisfactory with the exception of 2,3,7,8-TCDF which in early studies was observed to be uniformly low in comparison with those of the other compounds, most conspicuously with those of 2,3,7,8-TCDFs. A reexamination of the elution of 2,3,7,8-TCDF from alumina suggested that this step was the source of the problem; 2,3,7,8-TCDF eluted very early at the collection cutoff point. The addition of 5 mL of elution volume increased the recovery of [2,3,7,8-TCDF] to levels comparable with those of the other compounds.

Determinations of background levels of PCDDs, PCDFs, and non-ortho PCBs were routinely made as part of the control protocol. Procedural blanks and samples of laboratory-reared fish, each spiked with the marker compounds, were incorporated at a frequency of about 10% in all sample sets. Analyses of these control samples were used to define the background level for sample sets and to assess possible residue carry-over among samples. Of 14 procedural blanks, 1 produced a positive determination for OCDD at 1.6 ppb, 7 were positive for OCDD (1, 5, 7, 9, and 11 ppb), 1 was positive for a 2,3,7,8-TCDF at 2 ppb, and 2 were positive for OCDF at 0.5 and 1.4 ppb. All results for the 10 congener groups (total of 140 determinations) in these procedural blanks were negative and were below an average lower limit of detection of approximately 2 ppb. Of 11 analyses of samples of laboratory-reared carp, 7 produced positive determinations for OCDD (1, 1.5, 2, 3, 3, 3, and 6 ppb), 1 was positive for OCDF at 4 ppb, 1 for a HCDF at 2 ppb, 3 for a HpCDF at 2 ppb, and 5 were positive for OCDF (1, 1, 2, 3, and 4 ppb). The remainder of the 110 determinations of OCDD and PCDFs in these control fish were negative. The limit of detection was approximately 2 ppb. Non-ortho PCBs were not observed in these control samples, and the limit of detection for these compounds was approximately 5 ppb. In one series of control samples of laboratory-reared trout, a number of PCDF isomers were detected at 10–20 ppb levels. These compounds were identified as trace contaminants in the commercial fish and in the rearing.

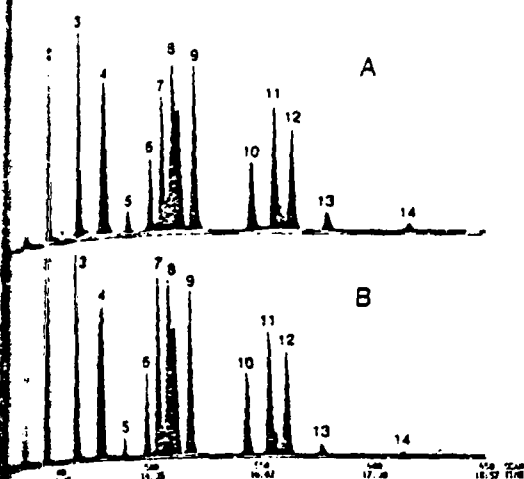
Background levels of PCDDs, PCDFs, and non-ortho PCBs were negligible, especially for those isomers having the 2,3,7,8-substitution pattern. Octachlorodibenzo-*p*-dioxin appears to be a common trace environmental

contaminant, being detected in more than 50% of the fish samples at levels significantly above those observed in the procedural blanks.

Although repeated analyses of procedural blanks between sample sets established a nondetectable level of carry-over between biological samples containing widely varying concentrations of PCDDs, PCDFs, and non-ortho PCBs, sample cross-contamination (from a carbon column) was observed to result from certain types of samples containing abnormally high levels of these contaminants. The samples causing cross-contamination were pond and river sediments and a sample of Aroclor 1260, all containing relatively high concentrations of PCDFs. Carry-over of PCDFs was readily demonstrated to result from reuse of the carbon columns and was observed in samples of fish which were processed on the same carbon column used for the highly contaminated samples. The degree of carry-over appeared to be on the order of 0.1%. In general, procedural blanks should be incorporated in sample sets at a frequency which will permit early detection of carry-over problems and should be included immediately following samples suspected of containing abnormally high concentrations of PCDDs, PCDFs, and non-ortho PCBs. Particularly in the case of sediment samples, high levels of other types of contaminants are routinely encountered, especially polynuclear aromatic hydrocarbons, and saturation of the carbon adsorbent with these substances may contribute to the problem of carry-over of PCDDs and PCDFs. In two cases of gross contamination of the carbon adsorbent, repeated washings of the column did not completely eliminate the problem, and the columns were replaced.

A satisfactory and reproducible level of recovery for 2,3,7,8-TCDD having been established, the recoveries of the other 21 TCDD isomers were examined. The mass chromatograms of a mixture of the 22 TCDD isomers (mixture provided by Dr. H. R. Buser, Swiss Federal Research Station, Wädenswil, Switzerland) before and after having been subjected to the enrichment procedure are presented in Figure 4. The relative recovery data, normalized to the recovery of 2,3,7,8-TCDD, are given in Table III. These data, although not rigorously demonstrative of satisfactory recoveries for each of the other 21 isomers, do establish that most of these isomers were effectively recovered by the procedure. In fact, in this experiment all other isomers or groups of isomers were apparently recovered more efficiently than was 2,3,7,8-TCDD. The abnormally high calculated recoveries of the 1,2,6,8-, 1,2,6,7-, and 1,2,8,9-TCDDs, each a minor component of the mixture, are attributed to the disproportionate influence of variations in instrumental sensitivity on analyte response near the limit of quantitation.

Probably the most useful piece of information derived from an examination of the determinations of the marker compounds in the hundreds of samples was the fact that the success rate for analyzability of the samples was better than 99% and that the minimum level of detection consistently



GC/MS-MID electron impact ion chromatograms of 22 samples: (A) following application of enrichment procedure; (B) before enrichment.

range of 1–10 ppt with an average value of less than 1 ppt. Samples and controls were routinely spiked at the 10 ppt level with each of the marker compounds. In all cases, the positive and uniform responses of the marker compounds in each of the analytes, GC/MS results for TCDDs and PCDFs at low parts-per-trillion levels were readily attainable. Estimates of the lower limit of detection (LOD) for TCDDs, TCDFs, and OCDD were made from the observed signal-to-noise value for the marker compounds (internal standards) to the LOD corresponding to a signal-to-noise value of 3. LODs require comparisons of the noise levels in each group of compounds and appropriate estimates of the internal standards.

Determination of PCDDs, PCDFs, and non-ortho PCBs is increasingly difficult at levels approaching the limit of detection, particularly to increased variations in the isotopic components of the molecular ions. Determination of the correct isotopic abundance ratios of molecular ions in determinations of PCDDs and PCDFs at low parts-per-trillion levels was usually the most difficult to meet once sufficient instrumental sensitivity was obtained. Nevertheless, over 50 separate confirmations made of PCDD and PCDF residues present at 10 ppt. The criteria for the confirmation of any PCDD, PCDF, or non-ortho PCB of unspecified substitution were: (1) signal-to-noise ratio of  $\geq 3$ ; (2) correct molecular mass; (3) coincidental maxima of three or four scans of individual members of the molecular ion series; and (4) chlorine isotope ratios within 10% of the expected values for three to six members of the molecular ion series.

Confirmation of routine monitoring of the fragment ions of these characteristic loss of COCl from PCDDs and PCDFs was investigated and determined to be marginal for these ions. The criteria for confirmation of these ions also include a requirement of demonstrating unique relative retention time within 2–4 parts per trillion. For example, 2,3,7,8-TCDD is sufficiently resolved from other TCDD isomers on both a Silar 10C (31) and a DB-5 (32) capillary column to enable easy determination of acceptable limits for the variation in retention time of this isomer relative to that of the isotopic 2,3,7,8-TCDD. The retention time of 2,3,7,8-TCDD on the DB-5 column was also found to be sufficiently resolved from the 1,2,3,7- and 1,2,3,8-TCDD isomers that their presence could be observed

but would not produce a false-positive determination. The variation in the retention time of 2,3,7,8-TCDD relative to that of [ $^{13}\text{C}$ ]-2,3,7,8-TCDD on the DB-5 column was observed in numerous analyses of standard mixtures of the two compounds and found to be within 2 parts in 1000. All confirmations of 2,3,7,8-TCDD in samples analyzed by this procedure met this requirement and were often repeated on a Silar 10C column. Samples of particular importance were independently analyzed by other laboratories using complementary techniques such as high-resolution mass spectrometry or atmospheric-pressure chemical ionization mass spectrometry (53). Over 20 samples analyzed in this laboratory for PCDDs and PCDFs were subjected to independent analyses in other laboratories, including those of H. R. Buser (Switzerland Federal Research Station, Wädenswil, Switzerland) (54), Ronald Mitchum (National Center for Toxicological Research, Jefferson, AR) (55), Michael Gross (University of Nebraska, Lincoln, NB) (55), Robert Harless (USEPA, Research Triangle Park, NC), David Firestone (U.S. Food and Drug Administration, Division of Chemistry and Physics, Washington, DC) (56), John Ryan (Health and Welfare Canada, Food Division, Ottawa, Canada) (57), Patrick O'Keefe (New York State Department of Health, Albany, NY) (26), and Christopher Rappe (University of Umea, Umea, Sweden) (Table IV). The Columbia laboratory also participated in three interlaboratory studies of the effectiveness of different methods for the determination of 2,3,7,8-TCDD in fish. The agreement in both identification and quantitation between the results from this laboratory and those of the other laboratories was consistently good, and no false-positive results were indicated in any of the determinations made with this procedure (Table IV). In the majority of interlaboratory studies, the comparisons involved only determinations of 2,3,7,8-TCDD.

**Evaluation of Potential for Interference from Cocontaminants.** Determinations of PCDDs, PCDFs, and non-ortho PCBs in environmental samples at levels below 1 ppt are particularly susceptible to interferences and possible false-positive results as a consequence of the likely occurrence of a large variety of polychlorinated aromatic cocontaminants and because full-scan mass spectrometric analyses are usually unattainable. More than a dozen families of such compounds are recognized as potential interferences in these types of analyses (35, 58), including DDE and DDT and polychlorinated members of the following compounds: biphenyl (59), methoxybiphenyls (60), hydroxybiphenyls, diphenyl ether (61), methoxydiphenyl ethers, hydroxydiphenyl ethers (62), benzyl phenyl ether (63), naphthalene, biphenylene, phenylbenzoquinone (64), xanthene, and bis(phenoxy)methane (65). Most of these families of compounds have the potential to interfere with and produce false-positive results in determinations of PCDDs and PCDFs even in HRMS (35). The problem of interferences in determinations of PCDDs and PCDFs has been rigorously addressed experimentally in only a few publications (66), and in these was limited to a small proportion of the numerous families of potential interferences. Routinely, conclusions in regard to the potential for interferences in analytical procedures for PCDDs and PCDFs are made by inference from observations of the effectiveness of separation of comparable amounts of these interfering compounds from PCDDs and PCDFs, often with a relatively small number of isomers of these two families. For example, alumina has been shown to effectively separate PCBs from certain PCDD isomers (67). A more appropriate evaluation should include a large number of isomers of and a large excess concentration ( $10^4$ – $10^6$ ) of the potential interference relative to that of PCDDs or PCDFs.

As part of the validation of this procedure an evaluation was made of the degrees of interferences produced by seven



## Levels of Interlaboratory Studies and Comparisons of the Determination of 2,3,7,8-TCDD in Fish and Birds

levels of 2,3,7,8-TCDD reported (pg/g) at different laboratories								
	CNFR	no. 1	no. 2	no. 3	no. 4	no. 5	no. 6	no. 7 reported av
9						6	5	
47		67			77	89	67	
22		25			57	42	34	
117		113		b	128	99	183	
56		45	b	b	38	53	c	
96		100	b	b	107	199	178	b
USFDA <sup>d</sup>								
58		104	58	49, 58	<5	72	70	61
<1		<10	<1	<2, <2	<5	<2	<5	3.6
34		35	37	23, 32	51	25	33	30
38		45	33	19, 31	55	32	27	32
37		52	45	55				
36		39						
19		15	25					
<1		<9	<5	<25				
Independent Laboratories								
	CNFR		Swiss Fed Res <sup>f</sup>		Nat Center Tox Res <sup>g</sup>		Health & Wel Can. <sup>h</sup>	
herring gull, Lake Huron	160		165				132	
gull egg, Detroit River	70		75				80	
gull, Lake Huron	22, 27		29		10			
gull, Lake Erie	<1		5		<10			
lake trout, Lake Ontario	56, 58				54			
herring, control	<1				<10			
lake trout, Lake Huron	39				32			
lake trout, Lake Ontario	38				31			
gull, Saginaw Bay	94				75			
gull, Tittabawassee R., MI	81				65			

<sup>a</sup> Samples were not analyzed due to large amounts of materials in analyte. <sup>b</sup> Sample was lost. <sup>c</sup> Reference 50. <sup>d</sup> Reference 51. <sup>e</sup> MS EL. <sup>f</sup> HRGC/MS APL. <sup>g</sup> HRGC/HRMS EI.

of polychlorinated aromatic compounds (35). In the study were selected isomers of polychlorinated PCBs, naphthalenes (PCNs), diphenyl ethers, methoxybiphenyls (MEO-PCBs), methoxydiphenyl ethers (MEO-PCDPEs), hydroxybiphenyls (HO-PCBs), and hydroxydiphenyl ethers (HO-PCDPEs). The results demonstrate an upper limit to the level of interference from these individual compounds. The results demonstrate the ability of the procedure to effectively eliminate interference from all but a small number of PCN isomers and non-ortho PCBs present at concentrations of 100-500 000 times those of PCDDs and PCDFs. Levels of these compounds observed in environmental samples analyzed in this laboratory (68), but PCB isomers other than the non-ortho PCBs have not been observed in the analyses for PCDDs and PCDFs. Furthermore, the results suggest that the procedure is not susceptible to interference from 10 000 times the level of the other five families of compounds. About 10% of the compounds are recovered by the procedure and are observed in environmental samples but do not give positive determinations. Rarely, interference is observed in the procedure due to partial overlap of a Cl<sub>2</sub> isomer with the marker compound, [UL-<sup>13</sup>C]-2,3,7,8-TCDD. The effective elimination of numerous interfering compounds, such as DDE, known to be present in the fish samples which were analyzed by this procedure has been demonstrated by full-scan MS.

The procedure also recovers isomers of polychlorinated aromatic compounds. A large number of isomers of polychlorinated aromatic compounds were identified in this laboratory in a sample

of soot produced during an electrical accident involving the pyrolysis of PCBs in a state office building in Binghamton, NY, in 1982 (26, 69).

The only other group of polychlorinated aromatic compounds apparently observed in a small percentage of samples were the nonachloromethoxydiphenyl ethers. These compounds, of which there are three possible isomers, were tentatively identified in three fish samples, from Saginaw Bay (35, 68), the Housatonic River, and Chesapeake Bay. The presence of these cocontaminants in the analyte contrasts with studies of interferences which indicate that chlorinated methoxydiphenyl ethers would readily be separated from PCDDs, PCDFs, and non-ortho PCBs.

The presence of polychlorinated diphenyl ethers (PCDPEs) in the analyte can be especially problematic because these compounds often undergo fragmentation during electron impact MS by loss of two chlorines to produce mass spectra which are identical with those of PCDFs below the molecular ion of the diphenyl ether. Furthermore, the elution window of PCDPE congeners have been observed in this laboratory to overlap that of PCDF congeners possessing two less chlorine substituents, greatly increasing the possibility for false-positive determinations from GC/MS-MIM analyses. Monitoring of masses of the molecular ions of the PCDPEs, if practical, can essentially eliminate this possibility.

The susceptibility to interferences of these types of analyses is demonstrated by the results of an interlaboratory study conducted by the USFDA (56) of the effectiveness of six different enrichment procedures (for 2,3,7,8-TCDD) performed by six independent laboratories (see Table IV). The enriched samples were all returned to the USFDA laboratory for rigorous analysis. Of the seven sets of analytical results only two,

## Precision of Quantitation Using Internal Standards in GC/MS and GC/EC Analyses Before and After the Enrichment Procedure

	before enrichment procedure			after enrichment procedure		
	mean response by GC/MS <sup>a</sup>	% std dev by GC/MS <sup>a</sup>	% std dev by GC/EC <sup>c</sup>	% std dev by GC/MS <sup>b</sup>	% rel recovery by GC/MS <sup>b</sup>	% rel recovery by GC/EC <sup>c</sup>
	1.39	6	8	12	97	109
	0.54	17	5	8	96	113
	1.40	7	2	14	97	128
	0.05		2		160	129
	1.05	7		5	85	
	0.92	15	4	19	140	127
	1.36	19	10	9	80	107
	5.63	10	2	15	109	126
	1.60	9	4	16	113	150
	1.29	9	5	17	133	137
	1.18	5	5	17	143	150
	0.80	8	7	15	153	141
	0.97	11	4	20	135	157
	0.42	12	8	26	195	159
	0.31	26	7	36	177	114
	0.44	27	7	30	164	114
	0.18	5		5	117	
	0.88	10		13	103	
	0.97	6		14	78	
	1.00				100	
	0.66	18		18	115	
		11.9	5.1	16.3		
		9.8	4.9	14.1		

<sup>a</sup>PCDD, D = PCDD. <sup>b</sup>[<sup>13</sup>C]-2,3,7,8-TCDD used as reference compound. <sup>c</sup>2,3,7,8-TCDF used as reference compound.

that generated by this laboratory, were judged to be compromised by the presence of significant levels of natural or interfering substances. In fact, the presence of small amounts of superfluous substances in a number of samples prevented the determination of TCDD in 5 of 3 fortified samples, as indicated by quantitative analyses which were significantly greater than the levels of detection.

**Enrichment Procedures.** Quantitations of 2,3,7,8-TCDD, TCDF, and OCDD are made directly by comparison of the integrated responses of the native compounds with those of the isotopically enriched marker compounds. This is made by analysis of known amounts of the marker compound and an authentic quantitative amount of the native material under those GC/MS conditions used in analysis of samples.

During the first 2 years of use of this procedure, quantitations of other PCDDs, PCDFs, and non-ortho PCBs were made by the external standard technique using mixtures of approximately 12 compounds. Toward the latter half of 1982, quantitations of these compounds were performed using the major isotopic marker compounds as internal standards for all congeners. Usually [<sup>37</sup>Cl]-OCDD was used for quantitation of OCDD and OCDF, and [<sup>13</sup>C]-TCDD and [<sup>37</sup>Cl]-2,3,7,8-TCDF were used for quantitation of all other PCDDs, PCDFs, and non-ortho PCBs. Relative response factors for the various congener groups were determined by GC/MS analyses of mixtures of the marker compounds and a series of 20 synthesized PCDDs, PCDFs, and non-ortho PCB isomers.

An attempt was made to determine the suitability, in terms of accuracy and precision, of quantitations of all congener groups using the internal standards (isotopic marker compounds). The experiment involved GC/MS-MIM and GC/EC analyses (5 replicates each) of a mixture of 17 native PCDDs and the 5 isotopically enriched marker com-

pounds. This mixture was subsequently subjected to the enrichment procedure (5 replicates) and analyzed again by GC/MS-MIM and by GC/EC. The mean and standard deviations of the integrated responses of all compounds relative to that of [<sup>13</sup>C]-2,3,7,8-TCDD were determined by GC/MS, and 2,3,7,8-TCDF was used as the internal standard in GC/EC analyses (Table V). The level of variation as measured by standard deviation for GC/MS quantitations using the internal standard was twice that determined for the GC/EC analyses. The data indicate that GC/MS quantitations using TCDD or TCDF as an internal standard were significantly more precise for tetrachloro through heptachloro congeners than for OCDD and OCDF. In contrast, no such disproportionate trends in precision were observed in the GC/EC analyses. The large variations associated with OCDD and OCDF are believed to be in part a consequence of GC/MS instrumental problems which were being experienced at the time and not necessarily characteristic of these types of analyses. Analyses of the mixture following application of the enrichment procedure show that the mean standard deviation is increased but comparable to instrumental variation. Nevertheless, the results indicate an acceptable level of precision for GC/MS quantitations of Cl<sub>4</sub> through Cl<sub>7</sub> congeners using a TCDD or TCDF as an internal standard in samples subjected to the enrichment procedure.

Determinations of PCDDs, PCDFs, and non-ortho PCBs were routinely carried out in the electron impact GC/MS mode. The GC/MS-EI technique, in contrast to negative ion chemical ionization analysis, exhibits comparable sensitivity for the broad range of congeners and permits identification and quantitation of all components in a single analysis. Negative ion chemical ionization GC/MS (GC/MS-NICI) has been observed in this laboratory and elsewhere (70) to exhibit a markedly enhanced sensitivity to PCDFs relative to PCDDs and, generally, to the higher relative to the lower chlorinated congeners of both groups. The ability to determine tetrachlorodioxins and tetrachlorobiphenyls in particular suffers



GC/NICI, and consequently this technique is unsuitable for complete determination of PCDDs, PCDFs, and PCBs at part-per-trillion levels. On the other hand, GC/NICI is much less sensitive to background (especially from hydrocarbons) or cocontaminant substances and has yielded more easily interpretable data.

**Extraction.** The implicit assumption in using the internal standards incorporated at the beginning of the procedure is that the behavior of an isolated compound will be identical with that of the compound present in sample. This assumption is not valid for all enrichment processes except that of extraction of residues from the sample matrix. The extractability of bioincorporated contaminants in biological samples or sorbed residues from soils and sediments is particularly important in studies of PCDDs and PCDFs. Studies of the biochemistry of PCDDs and related compounds in mammalian systems have established that these compounds exhibit high affinities for a hepatic cytosol protein; conformation of some PCDDs, PCDFs, and non-ortho PCBs in biological samples may involve more than the conformation of these residues from solution in fatty deposits. It has been reported of the efficiency of extraction of PCDDs, PCDFs, or non-ortho PCBs. On the basis of comparisons of the results of interlaboratory studies (Table IV) involving a wide variety of extraction procedures used for identical samples of fish containing 2,3,7,8-TCDD have provided a reasonable estimate of the extractability of this substance from fish tissue. These studies suggest that the neutral column extraction procedure employed in this procedure is essentially equivalent to extractions involving complete digestion of the sample in concentrated aqueous base or acid. Such digestions are used to denature and hydrolyze all proteins and to liberate all intact TCDD residues. Referring to laboratory no. 1 in the USFDA study employed concentrated HCl; in the H&WC/USFDA study no. 3 employed digestion with KOH, and in study no. 7 employed digestion with HCl. Assuming that 2,3,7,8-TCDD is as strongly bound in these samples of fish as it is in any other PCDD, PCDF, or non-ortho PCB, the extraction procedure is expected to effectively recover residues of these compounds. The effectiveness of extraction could be species dependent and cannot be extrapolated to other animal systems without similar studies. Our rationale for addition of the internal standards to the samples at the beginning of the extraction procedure was that equilibration of the native residues with the internal standards could not be easily attained in the latter step. Consequently, losses in the homogenization and drying step are not included in the internal standardization procedure.

The adsorptive interaction of PCDDs, PCDFs, and PCBs with carbonaceous materials has been studied (71), and studies of fly ash containing these compounds have demonstrated that exhaustive extraction procedures are required (72). Consequently, a study was undertaken in this laboratory to determine the relative efficiencies of various methods of extraction of these compounds from sediment samples (73). The neutral column extraction procedure was compared with a procedure (72) which had been demonstrated to be effective for the recovery of PCDDs from fly ash. Although the results of the comparison were highly variable and no unambiguous determination of the relative efficiencies of the two procedures could be made, the results of the procedures was uniformly superior

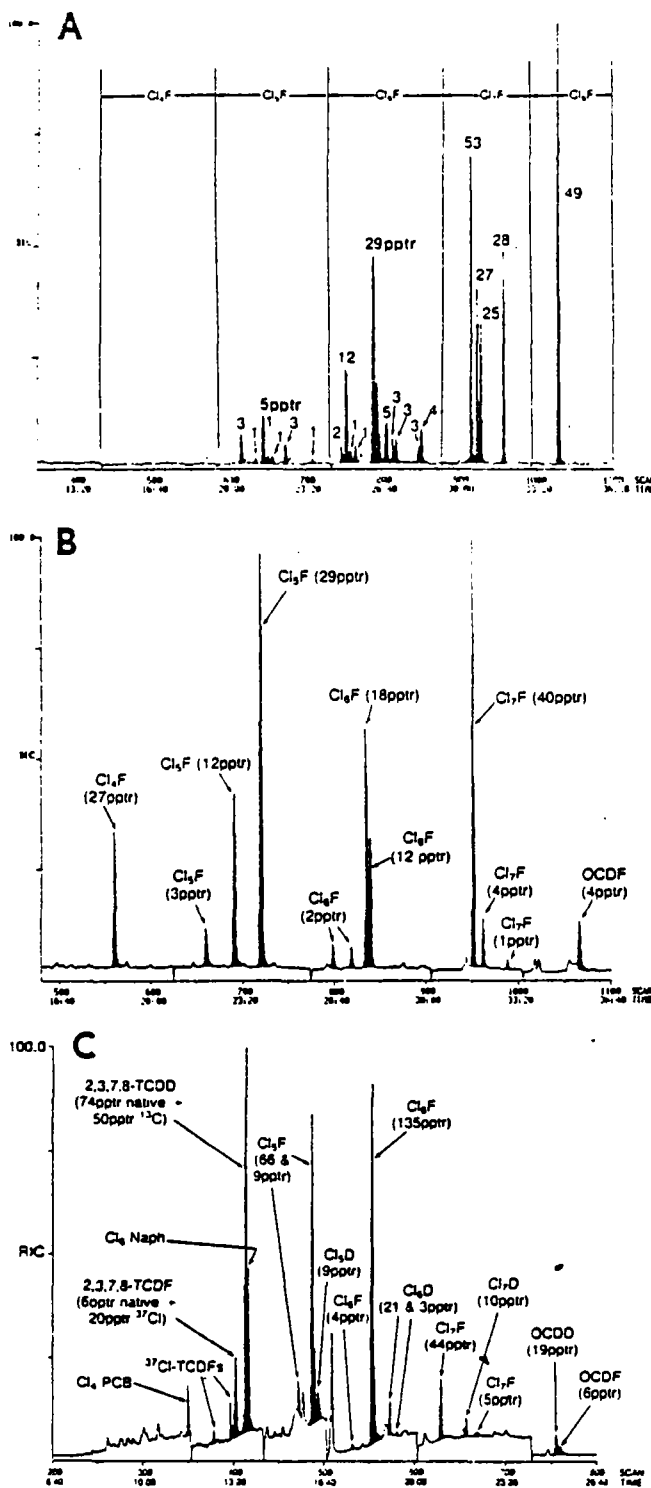


Figure 5. Representative analyses of environmental samples: (A) GC/NICI-MS-MID PCB contaminated soil from Fountain City, WI; (B) GC/NICI-MS-MID fish sample (carp) from Saginaw Bay at Bay City, MI; (C) GC/EI-MS-MID fish sample (carp) from the Niagara River at Ft. Niagara, NY.

to the other and appear to be roughly comparable in effectiveness. More definitive results are required from such studies before the efficacy of the column extraction procedure in analyses of soil and sediment samples can be established.

**Applications to the Analyses of Environmental Samples.** The procedure has been applied to the determination of PCDDs, PCDFs, and non-ortho PCBs in a wide range of sample types, primarily fresh-water fishes. The sample types which have been analyzed include about 12 species of fresh water fish (55, 68) and three species of salt water fish (both whole body and fillet): snapping turtle fat (54), whole body

approximately five species of fresh water mussels, made and eggs of three species of birds, Baltic aquatic macroinvertebrates, commercial fish and terrestrial soils (73), soot from an office involving PCBs and polychlorinated benzenes (26), and failed transformer fluid from a transformer. The large majority of these samples were collected on the five Great Lakes and selected tributaries of the Mississippi, Hudson, and Sacramento Rivers, and board rivers and estuaries, and the Housatonic in Massachusetts and Connecticut known to be contaminated with a wide range of persistent synthetic chemicals including organochlorine pesticides, and industrial wastes. A number of samples analyzed was approximately 50 control and procedural blank samples. Essentially all of the 250 analyses were judged to be acceptable according to the following criteria: (1) All marker compounds were detected in the analyte. (2) An acceptable detection limit (usually less than 5 ppb) was achieved. (3) GC/MS properties of analyte components were consistent. (4) PCDDs, PCDFs, and non-ortho PCBs did not show significant interferences. (5) The criteria for the detection of PCDDs, PCDFs, and non-ortho PCBs were

multiple ion mass chromatograms of soil samples are presented in Figure 5. These GC/MS analyses of PCDDs, PCDFs, and non-ortho PCBs in these types of samples serve to exemplify the procedure for such analyses. The GC/MS analyses were uncluttered by extraneous components, and the data was routinely straightforward.

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## Activity of Negative Ion Chemical Ionization Mass Spectrometry for Benzo[*a*]pyrene

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Gas chromatography/negative ion chemical ionization mass spectrometry (GC/NICIMS) was used as a selective and sensitive technique for the detection of benzo[*a*]pyrene (BaP). Under optimized conditions, the molecular anion,  $M^-$ , of BaP was more than 3 orders of magnitude more abundant than the isomer benzo[*e*]pyrene (BeP) using methane as the reagent gas. Quantities of BaP as low as 1 pg can be detected in the selected ion monitoring mode and the response vs. concentration was linear over a range of 3 orders of magnitude. The absolute sensitivity and the selectivity were found to depend on the pressure and temperature in the ion source of the mass spectrometer. GC/NICIMS was used for the quantitative determination of BaP, BeP, and benzo[*ghi*]perylene in a sample of petroleum crude oil as part of the process of certifying the Standard Reference Material.

Negative ion chemical ionization (NICI) mass spectra can be obtained from certain organic compounds by resonance transfer of thermal electrons if the molecules have positive electron affinities, and if the internal energy of the molecular ion is less than the electron affinity of the neutral species. The major species formed is the molecular anion,  $M^-$ , which yields relatively large ion currents and little fragmentation. The selectivity of NICI over electron impact ionization is well established and this feature has permitted wide applications over the past few years in the analysis of compounds such as polychlorinated biphenyls (1), pesticides (1, 4, 5), and nitrated polycyclic aromatic hydrocarbons (6). Iida and Dashima (7) recently reported the methane negative ion chemical ionization mass spectrometry of 21 polycyclic aromatic hydrocarbons (PAH). Oehme (8) determined PAH in air particulate matter using NICI. He used a mixture of methane and nitrous oxide as the reagent gas for negative ionization by electron capture and ion/molecule reactions and was able to differentiate isomeric PAH by the relative abundances of various species formed. Clapek, and Cooks (9) used negative ion chemical ionization mass spectrometry as a highly sensitive means for determining polycyclic aromatic hydrocarbons in a solvent refined coal. We have used NICI mass spectrometry as a sensitive and selective technique for the quantitative determination of

benzo[*a*]pyrene (BaP) in a sample of petroleum crude oil which is being certified as a Standard Reference Material (SRM). During the course of preliminary studies we have confirmed the large degree of selectivity for the detection of BaP over benzo[*e*]pyrene (BeP) noted by others (7, 8). We have observed the molecular anion of BaP to be more than 1000 times more abundant than that of BeP under selected source conditions in the NICI mode using methane as the reagent gas. Our observations, reported here, show that the ion source pressure and temperature play an important role in the selectivity of detection for BaP. We have also observed excellent absolute sensitivity for the detection of BaP and are able to detect quantities as low as 1 pg in the selected ion monitoring mode.

### EXPERIMENTAL SECTION

Negative ion chemical ionization mass spectra were recorded on a Hewlett-Packard 5985B quadrupole GC/MS system (Hewlett-Packard Co., Palo Alto, CA) with a dual EI/CI ion source and electronics capable of detecting negative ions. Chromatographic separations were carried out on a 30 m  $\times$  0.25 mm i.d. fused silica capillary column coated with a 0.25- $\mu$ m film of a nonpolar liquid phase. Samples were injected in either the split or splitless modes with an injection port temperature of 300  $^{\circ}$ C and the column temperature was programmed from 200 to 300  $^{\circ}$ C at a rate of 4  $^{\circ}$ C/min. The column was interfaced directly to the ion source by inserting it through a 30 cm length of 0.16 cm o.d. stainless steel tubing. The stainless steel tubing also served as a conduit for introduction of the methane reagent gas (Matheson Ultra High Purity 99.97%) which was brought in coaxially with the capillary column. The pressure in the ion source was adjusted by varying the methane flow into the source via a flow controller. An ionization gauge, which was mounted approximately 15 cm from the source, was used to monitor the ion source manifold pressure. The pressure in the ion source itself was measured with a thermocouple gauge. Spectra were recorded under conditions optimized empirically for the detection of BaP. The ion source was normally operated at 200  $^{\circ}$ C with a filament emission current of 300  $\mu$ A and a primary electron beam energy of 60 eV. The mass spectrometer was calibrated in the NICI mode using ions at  $m/z$  414, 452, and 633 from perfluorotributylamine and ions at  $m/z$  233 and 235 from rhenium oxide generated by the filament. The  $ReO_3^-$  isotopes provide a good source of ions at low mass for tuning the mass spectrometer in the negative ion mode.

The PAH were obtained commercially: BaP (Community Bureau of Reference, BCR, Brussels, Belgium); BaP- $d_{12}$  98.6 atom % D (MSD Isotopes, St. Louis, MO); and BeP (Pfaltz and Bauer, Inc., Stamford, CT). The standards were analytical grade or higher and were used without further purification. Methylene chloride solutions of the PAH were prepared gravimetrically. The Wilmington crude oil sample was obtained from the Department of Energy and is one of the oils being stored in the EPA Repository

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